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PREFACE

Use of the first edition of the "*Soil Sampling Quality Assurance User's Guide*" as a text in a series of seminars conducted at various U.S. EPA Regional Offices elicited many constructive comments for improvements from seminar attendees. Many of these suggested improvements have been incorporated in this second edition.

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CHAPTER 1

INTRODUCTION

OBJECTIVES

This document is a user's guide presenting and explaining selected principles and applications of methods and procedures for establishing adequate quality assurance on soil sampling aspects of environmental monitoring programs. Soil sampling aspects treated include sample site selection, sample collection, sample handling, sample analysis, and interpretation of resulting data. No detailed treatment of analytical quality assurance procedures is given, since that important aspect has been adequately treated elsewhere (U.S. EPA, 1982; U.S. EPA, 1984b). It should be noted, however, that sampling quality assurance procedures are not separable from analytical quality assurance procedures. This is particularly true for sample collection and handling. If an intact, timely, and representative sample of proper size and composition is not delivered to the analytical laboratory, the analytical methods and associated quality assurance procedures cannot yield meaningful results. Thus, the soil sampling quality assurance procedures presented here should be viewed as an important, integral part of the overall quality assurance plan.

In this second edition of the Soil Sampling Quality Assurance User's Guide, the authors have included guidance on developing Data Quality Objectives (DQOs) and have added additional examples to aid the user in preparing an adequate soil sampling quality assurance plan. This guide is not intended to be a generic plan for all sites; it is presented as a guidance document only. By adhering to the principles and procedures outlined, the user should be able to develop a quality assurance plan that will meet most soil sampling needs.

AUDIENCE

This document has been developed to serve as a user's guide for anyone designing, implementing, or overseeing soil monitoring programs. It is especially applicable for personnel responsible for regulatory programs where soil monitoring is an important integral element. Special attention is given to soil sampling examples related to CERCLA, since such applications are deemed to be high priority sampling programs. Many of the principles and procedures discussed, however, are applicable to other situations as well.

APPROACH

Following the discussion below of the background of quality assurance procedures used by the U.S. Environmental Protection Agency (U.S. EPA), Chapter 2 addresses purposes for soil sampling and Chapter 3 addresses the development of DQOs for various aspects of soil sampling. Chapter 4 presents an outline of the objectives of quality assurance plans. Chapters 7 through 10 deal with statistical aspects of experimental design, soil support, quality assurance/ quality control (QA/QC), hypothesis testing, etc. Some attention will be focused on both geostatistics and a technique known as the components of variance analysis. The components of variance analysis results from the use of a statistical sampling plan designed to measure as many of the sources of variation as can be identified and sampled in a cost-effective manner. The analysis further identifies the amount of total sample error (or variance) that results from each component in the sampling-analysis chain.

Discussion of the value of an exploratory study (Chapter 6) to the subsequent design of a soil sampling quality assurance program leads logically into more detailed discussions of sample site selection, sample collection, and sample handling (Chapter 11). These detailed discussions will include minimal coverage of soil monitoring protocols *per se*, since they have been treated in a comprehensive document (Mason, 1983). The focus of the discussions will be quality assurance. The goal of each discussion will be the development of design features for sample site selection, sample collection, and sample handling to meet quality assurance objectives of defined power and levels of confidence for each subject area.

The goal of the discussion concerning analysis and interpretation of data (Chapter 12) will focus on quality assurance aspects. The goals of the discussion concerning analysis and interpretation of data program audits and personnel training are treated in Chapter 13. To the maximum extent feasible throughout this report, we will first present concepts and principles, followed by selected examples of how these concepts and principles may be applied in realistic situations.

BACKGROUND

Since its founding, the U.S. EPA has been aware that the environmental data needs of the Agency require that quality assurance and quality control (QA/QC) meet predetermined standards. U.S. EPA Order 5360.1 (U.S. EPA, 1984) establishes the responsibilities of National Program Managers in the Agency's Mandatory Quality Assurance Program. These responsibilities include ensuring that "data quality acceptance criteria" and QA Project Plans are prepared for all data collection projects sponsored by the Agency. In a memorandum of

April 17, 1984, accompanying the issuance of Order 5360.1, Deputy Administrator Alm identified two steps that must be taken to ensure that all data collected by the U.S. EPA are suitable for their intended use:

> "...the user must first specify the quality of data he needs; then the degree of quality control necessary to assure that the resultant data satisfy his specifications must be determined."

The first step is accomplished through the development of Data Quality Objectives (DQOs). Data Quality Objectives are qualitative and quantitative statements developed by data users to specify the quality of data needed from a particular data collection activity (U.S. EPA 1987a). DQOs must address five data characteristics: precision, accuracy, representativeness, completeness, and comparability. A sixth data characteristic, level of detection, should also be addressed since it is closely related to the other five. In addition, DQOs should specify allowable probabilities of false positive (Type I) and false negative (Type II) errors. In order to determine required numbers of samples, another important factor is the desired minimum detectable relative difference between two data sets taken at different locations or times. The data quality characteristics addressed are sometimes referred to as measurement DQOs, while the probabilistic goals are termed system DQOs.

The second step in the QA process is the preparation of quality assurance project plans (QAPPs). The QAPP addresses the procedures to be followed to assure that the needs expressed by the DQOs are met. The DQOs become plumblines against which the data generated by a sampling effort can be evaluated. The whole quality assurance process is carried out to insure that the regulator, decision maker, or researcher has reliable data of known quality.

The chapters that follow address the various steps required to assure the quality of soil sampling data.

CHAPTER 2

PURPOSES FOR SOIL SAMPLING

The mission of the U.S. EPA is to control environmental pollutants to abate potential adverse effects on man and/or the environment. Complying with this mission requires identifying significant sources of pollutants of concern and linking these emission sources to adverse effects upon critical receptors. This linking is done through exposure assessment. To carry out the intent of CERCLA, for example, concentrations of hazardous pollutants in environmental media, including soils, should not be allowed to exceed levels established as being adequately protective of humans and the environment. Identification of the sources of the pollutant of concern should include not only the present emissions but also an assessment of probable future emissions. From a soil perspective, one needs to establish the role of soils as sources or sinks for selected air or water pollutants and how that role may change in time and space, as well as the effect of such physical parameters as temperature, wind direction and speed, water flow rates, and geological factors on that role. Biological factors within the soil matrix may also be involved in the degradation or transformation of pollutants into different chemical substances.

Specifically, soil sampling efforts can be designed and conducted to:

- determine the extent to which soils act as either sources or sinks for air or water pollutants,
- determine the risk to human health and/or the environment from soil contamination by selected pollutants,
- determine the presence and concentration of specified pollutants in comparison to background levels,
- determine the concentration of pollutants and their spatial and temporal distribution,
- measure the efficacy of control or removal actions,
- obtain measurements for validation or use of soil transport and deposition models,
- determine the potential risk to flora and fauna from specific soil pollutants,
- identify pollutant sources, transport mechanisms or routes, and potential receptors,
- contribute to a research technology transfer or environmental model development study, and

- meet the provisions and intent of environmental laws such as the Resource Conservation and Recovery Act (RCRA), the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), the Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA), and the Toxic Substances Control Act (TSCA).

Soils encompass the mass (surface and subsurface) of unconsolidated mantle of weathered rock and loose material lying above solid rock. The soil component can be defined as all mineral and naturally occurring organic material 2 mm or less in particle size. This is the size normally used to distinguish between soils (e. g., sands, silts, and clays) and gravels. In addition, the 2-mm size is generally compatible with analytical laboratory methods/capabilities. Organic matter is commonly found in many soils and must be considered as an integral part of the soil.

The non-soil fraction (e.g., automobile fluff, wood chips, various absorbents and mineral/organic material greater than 2 mm) must also be addressed during the sampling effort. This component may contain a greater amount of contaminant(s) than the associated soil. At sites in which this occurs reporting contaminant levels only in the soil fraction will ultimately lead to inappropriate and incorrect decision making. Decision makers must be aware that a number of problems are normally encountered in obtaining and using data from non-soil components. For example, questions arise concerning the validity of data obtained from the analysis of materials that do not meet the size and volume requirements in which the analytical processes were validated. Also, standard reference and audit materials are not available to substantiate and validate analytical results. The current recommended procedures are to identify and record the type and volume of non-soil material for each sample collected. A minimum of 10 percent of these non-soil samples should be submitted for analysis. Proper data assessment and conclusions made from these results are paramount to the success of a soil sampling program.

The behavior of pollutants in the soil environment is a function of the pollutant's and soil's physical and chemical properties. Soil sorption (the retention of substances by adsorption or absorption) is related to properties of the pollutants (e.g., solubilities, heats of solution, viscosity, and vapor pressure) and to properties of soils (e.g., clay content, organic content, texture, permeability, pH, particle size, specific surface area, ion exchange capacity, water content, and temperature). The soil components that are most associated with sorption are clay content and organic matter. The soil particle surface characteristics thought to be most important in adsorption are surface area and cation exchange capacity (CEC).

The extreme complexity and variability of soil necessitates a multitude of sampling/ monitoring approaches. The investigator must select methods and approaches that will satisfy the stated program objectives while accommodating specific site needs.

Both field and laboratory tests are necessary to understand the presence and behavior of pollutants in soil. Field tests primarily supply definitive information for soil classification and its relation to on-site environmental conditions. Laboratory tests supply analytical data beyond the capabilities of most field measurements, such as the type and quantity of a pollutant.

Soil containment measurements may be source-, transport-, or receptor-oriented, or some combination. For example, if the major concern is possible risk to human receptors, it may be wise to take early measurements in the immediate vicinity of the receptors to obtain the best estimates of exposures resulting from soil contamination. If exposures are deemed to be insignificant or acceptable, no further measurements may be required. If, however, the exposures are deemed to be unacceptable, additional measurements will be required to identify both important pollutant sources and important exposure pathways. Information on these matters will be necessary to devise cost-effective control strategies.

Determination of risk to human health and the environment from contaminated soils involves several steps. Required are exposure and dose distributions to the most sensitive populations or receptors of concern via all significant exposure pathways. This will involve possible soil-related exposure from other media such as air or water, exposure from the soils themselves either through ingestion, inhalation, or skin absorption, as well as exposure through ingestion of foods contaminated directly or indirectly from the soils. An additional parameter is the biological availability of the pollutant(s) of concern. Thus, it is important to measure or estimate the extent to which the soils act as sources (through contacting air or waters) for the pollutant(s) of concern. Knowing the concentration of pollutants in air or water originating from contaminated soils is not sufficient for estimating exposure. An additional parameter required is the biological availability of the pollutant(s) of concern. For example, if soil pollutants are not incorporated into the edible parts of crops or animal products, even large concentrations in the soil might not lead to significant human exposure through ingestion of food stuffs. In such an instance, however, inhalation of vapors from the soil or ingestion of drinking water might constitute an important exposure pathway.

Once desired exposure or dose distributions have been constructed, comparison to established exposure or dose-response relationships enables a determination of whether or not the existing risk is acceptable. Underestimating exposures or doses might lead to accepting an unacceptable risk, whereas overestimating might lead to unnecessary, and possibly costly, control actions. A detailed case study showing how an action level for dioxin in soil was derived is presented in Appendix A.

If significant quantities of pollutant(s) become permanently attached to soil and remain biologically unavailable, the soils may constitute a sink. Pollutant control needs in these cases may be reduced by the amounts by which the soils reduce the pollutant availability. Underestimating the ability of soils to act as a sink might lead to source control requirements more stringent than necessary, whereas overestimating might lead to less stringent control requirements than necessary.

If significant quantities of selected pollutants are found to be associated with soils initially and then released slowly over relatively long periods of time, the soils, in essence, act as pollutant sources. Underestimating the extent to which soils act as sources will lead to inappropriate and insufficient controls of other additional sources, whereas overestimating may lead to expensive soil removal to a greater degree than necessary. Soil removal as a cleanup measure is a complicated proposition. It involves extensive testing of the soils and evaluation of proposed disposal options to determine which option will have the least environmental impact with due regard for cost.

Soil sampling to measure the efficacy of control or removal actions must be preceded by the establishment of unacceptable concentrations of pollutants of concern in soil. Once unacceptable concentrations, or action levels, have been identified, it is then possible to devise sampling plans with defined probabilities of Type I or Type II errors. A critical consideration in this instance will be the depth and surface areal extent of the soil sample on the basis of which the soil concentration will be calculated. This is addressed in greater detail in a subsequent chapter dealing with the concept of sample support and "action support" (Chapter 5).

Soil sampling for validation or use of soil transport and deposition models will not normally lead to control actions. Positive or negative errors are unlikely to lead to corresponding over- or underestimates of control needs. However, errors of unknown direction and size, if sufficiently large, might seem to validate an erroneous model or fail to validate an acceptable model. The consequences of such errors cannot be evaluated without knowing the purposes for which the model might be used and what actions might be taken on the basis of conclusions drawn from the model.

Prior to undertaking any soil sampling program to achieve defined objectives, it is necessary to establish appropriate measurement and system DQOs. These should be established after due consideration of the consequences of taking actions which might subsequently be shown not to be justified on the basis of the available data.

Once appropriate DQOs have been established, an operational protocol should be prepared, setting forth what is to be done for what purpose and how, when, where, and how many samples will be collected. Also, the protocol should indicate how the samples will be preserved, prepared for analysis, and then analyzed for what substances, and how the resulting data will be validated, analyzed, and interpreted. As part of this protocol, a complete QA/QC plan must be included covering all aspects of the experimental program with special attention to sampling aspects. Quality assurance is defined as the system of activities required to provide a quality product, whereas quality control is the system of activities required to provide information as to whether the quality assurance system is performing adequately. It cannot be overemphasized that an adequate QA/QC program cannot be tailored for a study until a clear statement of monitoring objectives has been provided, together with allowable errors.

CHAPTER 3

DATA QUALITY OBJECTIVES

Studies conducted in the past were often controlled by data quality that was considered to be the "best data possible" (U.S. EPA, 1986-b). It was not uncommon to expend considerable resources on a sampling and analysis program, only to find that the samples were not collected in a manner that would allow valid conclusions to be drawn from the resulting data. The "best data possible" approach provided useful data in some cases but frequently lacked the scientific rigor required for the regulatory arena. The development of Data Quality Objectives (DQOs) is an attempt to provide the rigor required to meet the data needs of the U.S. EPA.

"Data Quality Objectives are qualitative and quantitative statements of the quality of data needed to support specific decisions or regulatory actions" (U.S. EPA, 1986a). The important starting point for the detailed design of a data collection effort, DQOs are the basis for specifying the quality assurance and quality control activities and requirements associated with the data collection process. During the detailed planning and preparation of technical guidance for data collectors, DQOs are used as the key for developing explicit, quantitative statements of the type of errors that will be controlled, the level to which those errors will be controlled, and the information that will be collected in order to characterize all of the known sources of error. These quantitative statements are known as data quality indicators. Data quality indicators are needed to select appropriate methods for sample collection, laboratory analysis, and statistical data analysis. They also form the basis for selecting QA and QC procedures (U.S. EPA, 1987a).

The DQO process is a dynamic process that has not yet been implemented uniformly in all regions; therefore, the information presented in this document is for guidance only. The three-stage process envisioned in guidance documents (U.S. EPA, 1986a, 1987a) includes requirements for the following factors to be addressed:

- precision,
- accuracy,
- completeness,
- representativeness, and
- comparability.

A sixth factor, "detection limit," has been added by the authors as a critical factor which should be considered in specifying the other five factors.

Statistical rigor, combined with managerial and budgetary guidance, should be used to develop the specific objectives required to develop the specifications for the above factors. Statistical sampling is a mechanism by which the QA/QC program can determine the sampling precision and provide a measure of the reliability of the entire sampling effort.

It is essential that the reliability of the data be reported. Buffington (1978) quotes Congressman George E. Brown, Jr., as saying "no number is significant, and subsequently worthy of being recorded, without an estimate of its uncertainty." This statement should be considered when designing the QA/QC plan for a soil sampling effort because soil is by its very nature extremely variable. Superimposed on this natural variability are other sources of variation or error that can be introduced into the final result by the sampling and analytical efforts. These sources of variation can lead a manager to conclude that an area needs no remedial action when, in fact, it does need such action (called a Type II error) or, alternatively, conclude an action is needed when, in fact, no actions should be taken (called a Type I error).

To establish an adequate, cost-effective QA/QC plan for a soil monitoring program, it is necessary, after careful analysis of the consequences, for a decision-making official to specify probabilities of Type I and Type II errors that will be allowed in making decisions based on sample data.

The acceptable probability for each type of error must be established in relation to the consequences of making such errors and depends upon the specific objectives of the soil monitoring program. The Type I error is the error most often considered in the literature. In environmental monitoring, however, a Type II error may be more important than a Type I error. The clean-up of a highly toxic spill would be an example where a false negative could create major problems for the project manager. The Type II error would lead the manager to conclude that a clean-up of some areas is not necessary when, in fact, the action levels are being exceeded and clean-up is necessary. The probabilities of Type I and II errors for the QA/QC effort should equal the probability levels chosen for the overall sampling effort itself. This acceptable probability of error in different cases may, for example, range from 20 percent to 1 percent or less. In some circumstances, the level selected by value judgment may simply be a statement of a probability of error not to be exceeded in the final data. The authors are proposing that these two probabilities be included as system DQOs in conjunction with the measurement DQOs outlined earlier, i.e., precision, accuracy, representativeness, completeness, comparability (PARCC), and detection limit.

There may be a temptation in many cases to avoid making the necessary value judgments concerning acceptable probabilities of making different kinds of error. The course of action often substituted for the difficult value judgment is to adopt as a guiding principle the concept that one should always strive to achieve the highest power and level of confidence (or lowest probability of error) possible with existing available resources. The resulting data are then used as the basis for making decisions with the assumption that this guiding principle gives the best possible result. Obviously, such an approach will rarely, if ever, be cost effective. Two types of errors are possible. The data may be much better than required, which indicates resources have been wasted, or the data may not be of adequate quality, thereby resulting in decisions of doubtful validity. This point may be summarized by stating that resource availability is an important factor for consideration in the establishment of quality assurance programs, but resource availability should not be accepted as the sole determinant of required quality assurance methods and procedures. Maximal cost effectiveness should be the overall goal. This generally means that a minimum adequate quality assurance plan must be defined and then implemented. The DQO process has been designed to incorporate both cost and reliability of the data as a first principle, and thus precludes the tendency to use cost as the single guiding principle for implementing a particular sampling design.

Once system and measurement data quality objectives (DQOs) have been set on the basis of acceptable risks of making mistakes in any resulting decisions, an experimental design can be adopted to achieve the required DQOs. The purpose of the Quality Assurance Project Plan (QAPP) then becomes the collection and analysis of adequate QA/QC samples to confirm the achievement of the DQOs. Another way of stating this is that the objective of the QAPP is to define the procedures used to achieve the desired quality of the data and thus insure that it is adequate to the degree required for its intended end use. (These matters will be covered more extensively in subsequent chapters).

Guidance on the DQO process (U.S. EPA, 1987a) identifies three stages for arriving at the quality of data to be used:

1) Identify decision types,
2) Identify data uses/needs, and
3) Design data collection program.

Figure 1 identifies each stage along with the tasks covered in each stage. Thus, the DQO process becomes an iterative interaction between management and the technical staff. Management identifies the needs and resources available. The technical staff develops guidance for assisting management in making the decisions required to develop the DQOs. The end result is site-specific guidance for evaluating and interpreting sampling data.

Agency guidance (U.S. EPA, 1987a) provides the basis for developing the DQOs. Statistical designs should be used so that the quality assurance procedures can verify that the DQOs are being met. Where possible, numerical values or limits should be placed on precision, accuracy, representativeness, completeness, comparability, and detection limits. Once these numerical measures are defined, the preparation of statistical designs, sampling protocols and quality assurance plans can be initiated. The result of such a process meets the needs of the Agency for quality data, addresses the requirements of the user, and aids the technical staff in providing the quality of services requested.

Chapters 7 through 10 discuss in detail the statistical aspects of sample design and data evaluation.

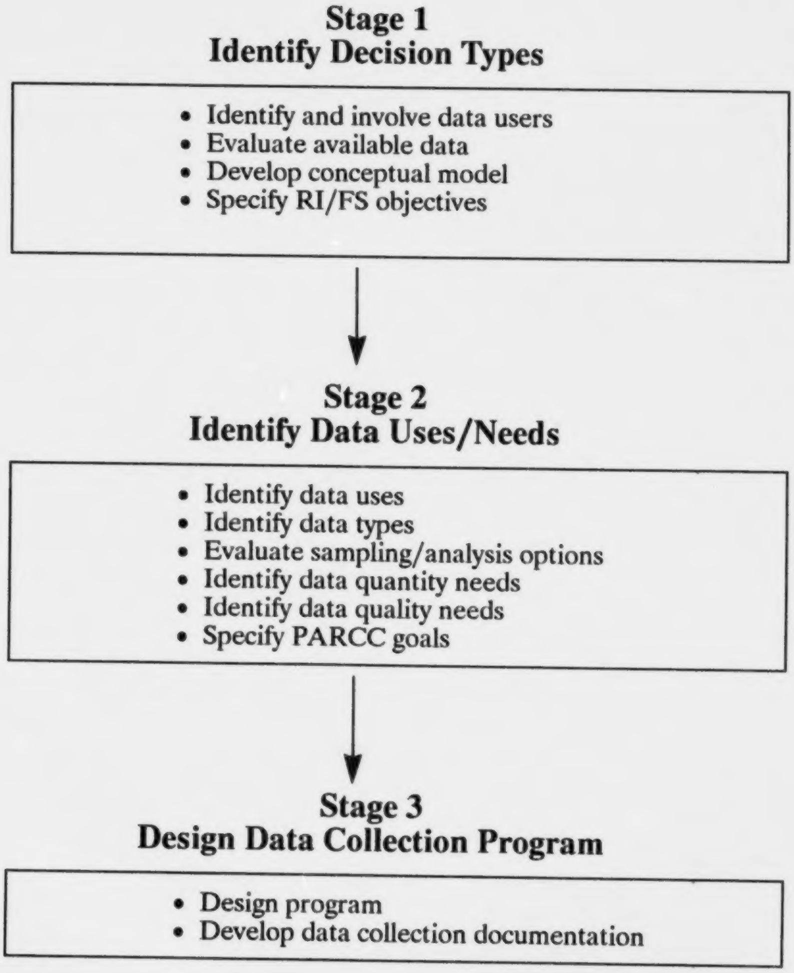
Stage 1
Identify Decision Types
• Identify and involve data users
• Evaluate available data
• Develop conceptual model
• Specify RI/FS objectives
Stage 2
Identify Data Uses/Needs
• Identify data uses
• Identify data types
• Evaluate sampling/analysis options
• Identify data quantity needs
• Identify data quality needs
• Specify PARCC goals
Stage 3
Design Data Collection Program
• Design program
• Develop data collection documentation

Figure 1: Three Stages of the DQO Process (U.S. EPA, 1987a)

The soil sampling requirements of both CERCLA and RCRA are site-specific. A history of the site, including the sources of the pollutant and a conceptual model of the routes of exposure should be developed before the sampling plan is finalized. It may be necessary to conduct an exploratory study before this conceptual model can be confirmed or a different model defined. (Chapter 6 outlines guidance for conducting the exploratory study.) This study should provide insight into the types of pollutants present, the population at risk, and the magnitude of the risk. These factors can then be combined to design the final sampling plan and to specify the size of sampling unit or support (Chapter 5) addressed by each sample or set of samples.

MEASUREMENT CONCEPTS

The following three sections discuss some basic concepts that must be kept in mind when developing data quality objectives for soil sampling. These concepts may have application in other types of sampling, but they are considered to be particularly pertinent to soil sampling efforts.

Support

As presented in Chapter 5, the support for a sample is the unit of soil that the sampling effort measures. The support has a specific size, shape, and orientation. In the vernacular of the soil scientist, this unit of soil is not too unlike the pedon in terms of its dimensions and definition (Soil Survey Staff, 1975). The choice of support can significantly affect the precision of estimates obtained from the survey data (Starks, 1986).

Risk and exposure assessment data can be used to define an action level for a particular chemical. This action level must be defined as a concentration over a particular support. Starks (1986) identifies this particular support as an "action support."

The action support should be defined prior to establishing study objectives or, alternately, as part of the DQO system. The support must be kept in mind when characterizing acceptable levels of probability of Type I and Type II errors, when defining precision, when evaluating representativeness and comparability, and when defining detection limits for the analytical method used.

The Measurement Process

Data users often look at a concentration obtained from a laboratory as being "the concentration" in the soil without realizing that the number generated by the laboratory is the end point of an entire process extending from design of the sampling through collecting, handling, processing, analysis, quality evaluation, and reporting. Variation or error can occur at any of the steps in the process. The final number reported represents the actual concentration found in the soil plus a number of components of variation.

A regulator or researcher would like to have an analytical result that has no error in the reported concentration, but this is not possible to attain with a medium such as soil. Since it is not possible to eliminate the natural error in the measurement process, the investigator would like to know the variation so that he can use this information in making a decision or in controlling the quality of the data. A components of variance analysis provides a means for determining the source of the variation in the data and estimating its magnitude.

Examination of the results of a components of variance analysis performed on soils data from an NPL site sampled for PCBs indicates that 92% of the total variation came from the location of the sample, while only 8% was introduced after the sample was taken. Less than 1%

of the total could be attributed to the analytical process itself; yet, this latter area is where a majority of the QA resources are normally focused. These relative values are probably a reasonable pattern for many different soil studies. Properly specified data quality objectives should bring a balance into the QA/QC process.

Generally, a decision must be made as to whether a site is contaminated enough to cause an environmental problem, and, following this decision, within the site an area-by-area decision must be made as to which must be cleaned or remedied and to what extent.

No valid decision can be made about the data or the site under investigation without some knowledge of the magnitude and sources of error in the data. This aspect becomes very important when concentrations of pollutants in a support approach an action level. Concentrations that exceed the action level by orders of magnitude require only limited QA as do those areas that contain no pollutant. The area where sampling intensity and increased quality assurance becomes important are those areas where it is not possible to make a clear decision as to the need for and extent of action.

The design of a sampling effort and its associated quality assurance plan must accomplish three things:

- Determine the variability in the entire measurement process along with the sources and magnitude of the variation in the results generated;
- Provide a means of determining whether a sampling program meets the DQO's provided; and
- Identify areas of contamination where action is needed.

STAGES FOR DEVELOPING DQOS

Figure 1 shows the three stages for developing the data quality objectives for a study or decision process. Each stage is discussed below.

Stage 1: Identify Decision Types

Stage 1 of the DQO process provides the foundation for Stages 2 and 3. In Stage 1, all available information on the site is compiled and analyzed. Based on the available information, a conceptual model or models (related to different categories of pollutants present) of the site are developed. These models describe suspected sources, contaminant pathways, and potential receptors. The models will assist in identifying decisions which must be made as well as deficiencies in the existing information. Stage 1 is undertaken to define the types of decisions which will be made during the remedial investigation/feasibility study (RI/FS) and involves defining program objectives and identifying and involving end-users of the data. The decision maker and all potential data users should be involved in this and all subsequent DQO stages. Stage 1 results in the specification of the decision-making process and justification for the collection of any new data (U.S. EPA, 1987a).

Identify and Involve Data Users: The person with primary interest in the DQO process is the ultimate decision maker. It is not likely that this individual will be directly involved with the process of developing the DQOs, but a representative will be designated by him to help make the necessary detailed decisions. Individuals likely to become involved are:

- Regional Administrator or representative
- The Remedial Project Manager
- DOJ, EPA, and State Attorneys
- Chemists
- Quality Assurance Officer
- Statistician

- Risk specialist
- Other technical specialists needed to design and review the DQOs
- Consultants and contractors
- Other interested parties

The Remedial Project Manager (RPM) manages remedial activites and is accountable for the technical quality, schedule, and cost of the work. Therefore, he is the primary person responsible for insuring that the DQOs meet program needs for a particular site. The RPM is not expected to and should not develop the DQOs alone but must include the necessary engineers, hydrogeologists, soil scientists, chemists, statisticians, risk specialists, and toxicologists in the design of the DQOs.

In time, portions of the DQO development process may become standardized. Generic levels of measurement and system DQOs may be developed. Until such standard DQOs are established, the entire DQO development process will have to be followed in each case with all parties being involved. However, Regions may desire to establish a means for developing and reviewing a generic set of DQOs for use in emergency situations. This would avoid hasty decisions on the quality of data needed for clean-up and reduce the amount of time needed to field an emergency team.

There are occasions where the Potentially Responsible Parties (PRPs) may be asked to review the DQOs. This is usually addressed through the attorneys representing the PRPs and will be responded to by a consultant or a technical member of each PRP's staff. In cases where there is considerable public interest, representatives from interested parties also may be asked to review and comment on the DQOs. These reviews can greatly reduce the likelihood of conflict at later stages of the study.

The RPM must be familiar with the site to properly identify the potential data users. A site visit combined with the results of an exploratory study can provide a basis for selecting the individuals and disciplines to identify DQO requirements. The process of conducting the RI/FS is an ongoing, iterative process. As data become available, it may be necessary to redefine the team required to evaluate the data and provide guidance on the overall planning and execution of the sampling effort. Refinement of the data collection process also may require that additional technical staff be added to improve the review and evaluation of the data.

Chemists and statisticians should be a part of the planning for any soil sampling effort. The analytical chemist can provide insight into the types of analyses needed and the levels of

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Chemists and statisticians should be a part of the planning for any soil sampling effort. The analytical chemist can provide insight into the types of analyses needed and the levels of detection that are required to meet the objectives of the study. Soil scientists or geochemists should be included to aid in evaluating the interactions of the chemicals with the soil. Scientists and statisticians trained in geostatistics can provide guidance in the use of the various geostatistical tools to evaluate the spatial distribution of the chemicals.

The RPM may desire to have all of the various data users represented during the initial planning meetings. The choice of users and their involvement can be defined better after the first few meetings. Even though a user may not be directly involved in the development of the DQOs, all potential users should be given an opportunity to review and comment on the final study objectives, protocols, QA/QC plans, and reports.

Assemble and Evaluate Background Data:

Background data: Many sources of soil related data are available for use in planning a study. Mason (1983) outlines a number of sources of published and public domain soils data. A detailed list of other sources of background information is provided in an Agency document (U.S. EPA, 1985). Results of any preliminary or exploratory studies provide an excellent source of information for use in developing final DQOs. Data quality objectives for studies conducted in similar settings also provide an excellent resource for use in specifying DQOs.

The information that is being accumulated about a site for the first time will generally be more fragmentary and incomplete. The quality of the data at the outset may be insufficient to support required decisions. As the RI/FS efforts continue, the data should improve in reliability and become more useful in guiding further sampling efforts.

Site Visit: A site visit provides the basis for identifying the types of data that may prove to be useful to the team. When feasible, Agency team members as well as involved contractors should take part in the site visit. This visit may also provide information on any potential health and safety risks. Site visits are used to:

- Inventory other possible off-site sources of contamination;
- Identify any exposed populations;
- Confirm existing information;
- Record observational data about the site;
- Determine existing site conditions;
- Determine access, possible sampling points, obstructions, and site configurational limitations;
- Determine the possible presence of volatile chemicals, explosive hazards, etc.;
- Determine restrictions or limitations for particular RI activities;

- Delineate areas of waste storage or contamination and their contents;
- Determine the security of the site and identify where this needs to be repaired or improved; and
- Identify and document any monitoring, industrial or potable water wells on or near the site.

A topographic map at a scale large enough for recording changes to the site and other pertinent information such as locations of underground pipes or wires should be available during the site visit. Photocopies of portions of USGS, 7½-minute Quad sheets can be prepared. The graphic scale of the map should also be enlarged and copied at the same time as the map. This provides a means of plotting any objects or conditions observed.

A tool that can be quite useful for the site visit is a scaled aerial photograph of the site and adjacent environs. Information on obtaining and interpreting aerial photographs can be obtained at U.S. EPA's Environmental Monitoring Systems Laboratory, Las Vegas, NV.

Dated photographs and video movies of the site are also useful for preparing study protocols. These should be well documented so that they can be used as evidence at a later date if this becomes necessary.

All of these pieces of information become part of the official record to be assembled for the site.

Evaluate Existing Data: Data that have been assembled from all available sources should be evaluated as to their relevancy and accuracy. Adequate time should be spent in examining the data set that has been assembled. Often information that is not classed as valid because of QA restrictions can be used in establishing a hypothesis about how the pollutants on the site have behaved over time. These data cannot be used in making the final decisions about the need for clean-up, but they can be used to help develop a conceptual model for the site. Factors that must be considered in evaluating the data for their usefulness are:

- the age of the data sets and their comparability,
- the precision and accuracy of the data,
- the sampling design used to collect the samples,
- the methods used to collect, preserve, handle, and transport the samples,
- the analytical methods used to measure the pollutant,
- the detection limits for the methods, and
- the quality control measures used by the laboratory and field team.

Older data sets were often acquired by methods that are no longer considered to be valid. For example, soil samples for volatile chemicals were originally collected in a large container with headspace. Current methods call for use of a 125 ml wide mouth glass bottle filled with no headspace. Reported values for volatiles from the former method probably indicate only the qualitative presence or absence of volatile chemicals. The quantitative concentrations reported should be seriously questioned.

Similar to sample collection and handling methods, analytical methods have also improved over time. This should also be taken into consideration when evaluating previously collected data. Any uncertainty associated with the data should be a major consideration in evaluating the useability of the data. Keep in mind the discussion on sampling and analytical errors outlined in the section above on the Measurement Process. If a components of variance test was carried out or can be carried out on the data, the test results can be used to determine the usefulness of the data. One of the major factors that the investigator or RPM attempts to determine is an estimate of the probable precision and accuracy of the available data. Any available components of variance data can be used as a guide for designing the sampling effort to meet the DQO requirements.

The primary objective of the data evaluation process is to determine if the data are a valid representation of the site at the time the samples were collected. A valid representation of the site provides a means of determining if there have been changes in the conditions at the site over time. Some chemicals may have been degraded, volatilized or leached from the site. Other chemicals may have been deposited on the site, moved through or onto the site from surrounding areas, or formed at the site through various physical and biological interactions. This aspect of the evaluation becomes very important when litigation is anticipated.

Develop a Conceptual Model of the Site:

A guidance report (U.S. EPA, 1985) outlines procedures for developing a conceptual model or models. Essentially models are graphic and narrative descriptions of the site, the pollutants on the site, and the behavior of the pollutants over time. The models should describe all potential routes of exposure that may be important during the operation of the site and following deposition of the pollutants at the site. The graphic depiction of routes of exposure helps the RPM and the other decision makers to visualize where problems may exist. The models essentially become hypotheses that are to be tested by the sampling effort. A properly designed sampling plan will address all of the routes of exposure and the populations that may have been exposed. For example, vaporization, contact, and leaching are the major mechanisms whereby soil pollutants may be transported from contaminated soil to receptors. The vapors may be inhaled; soil particles may come in contact with skin, be inhaled, or be ingested; and leachates may become sources of surface and groundwater pollution. Figure 2, taken from U.S. EPA, 1987a, outlines the important elements of a conceptual model.

Investigators must be cognizant that soil is only a part of the total system that should be considered at a site. A model of the soil compartment includes the following components:

- soil cover,
- soil elevation contours,
- soil matrix,
- particle sizes,
- soil solution,
- soil vapor, and
- associated debris.

As collected data identify specific areas where the model is not valid, the model should be modified to reflect the new information. An important model input is the presence of contaminated non-soil debris in the soil mass which is usually one of the first things identified

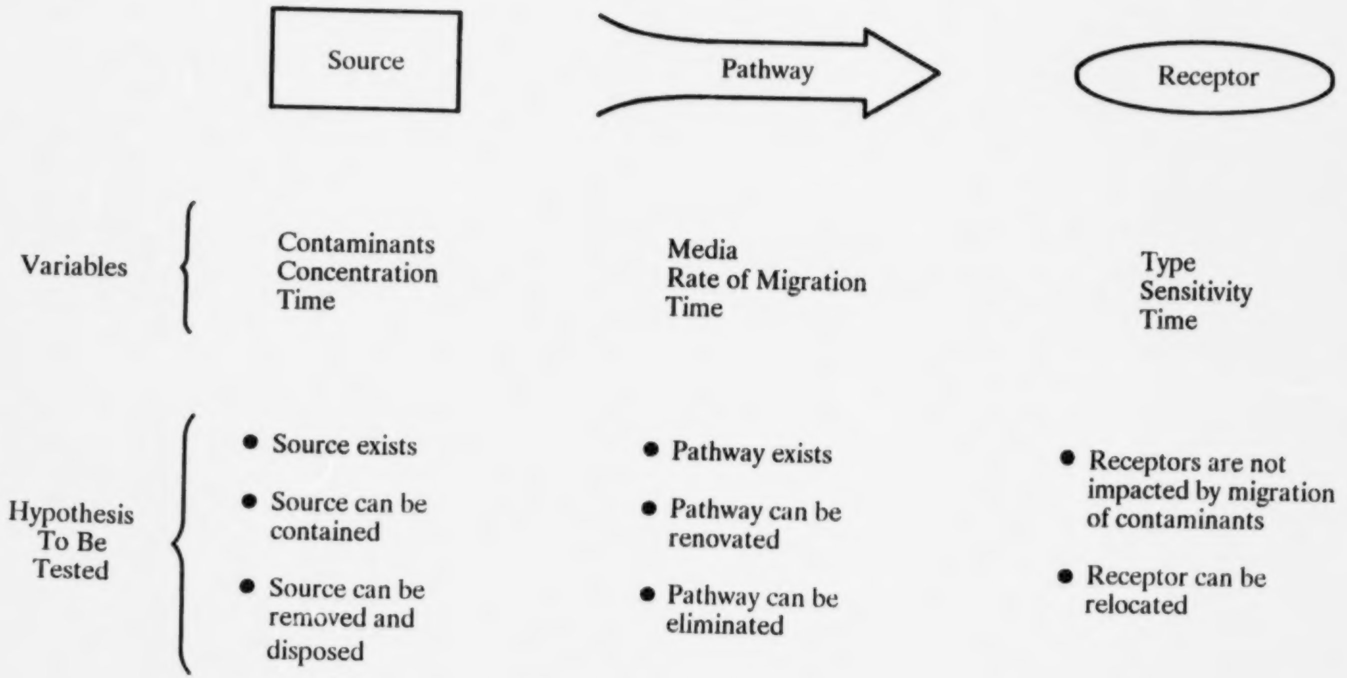
Source
Pathway
Receptor
Variables
Contaminants
Concentration
Time
Media
Rate of Migration
Time
Type
Sensitivity
Time
Hypothesis To Be Tested
Source exists
Source can be contained
Source can be removed and disposed
Pathway exists
Pathway can be renovated
Pathway can be eliminated
Receptors are not impacted by migration of contaminants
Receptor can be relocated

Figure 2. Elements of a Conceptual Model.

during soil sampling. This must be considered in the evaluation of the hazard posed by the site. Most soil sampling and analytical efforts attempt to remove large pieces of debris such as wood, etc., from the sample. In some cases, the debris rather than the soil may be the source of the pollution, since it is common to use wood chips or shredded wood as an absorbent for liquid wastes. Screening non-soil debris out of the soil material and excluding it from the analysis may bias the results against obtaining a valid assessment of the risk posed by the site.

Mathematical models or computer codes are often used to estimate the extent of the exposure. The conceptual model developed during the RI/FS can become the basis of the computer models used to evaluate the risk to exposed individuals. Modelers should become a valuable part of the team defining the DQOs for a particular site.

Specify the RI/FS Objectives:

The remedial investigation (RI) addresses data collection and site characterization to identify and assess threats or potential threats to human health and the environment posed by a site. The feasibility study (FS) identifies and evaluates remedial alternatives using appropriate environmental, engineering, and economic factors (U.S. EPA, 1987b).

Some of the questions that should be addressed before or during the RI/FS study include:

- Is the area contaminated with hazardous chemicals?
- What is the distribution of the chemicals over the site?
- Are there any areas that create a threat of an immediate life-threatening exposure?
- What are the dominant routes of soil exposure at the site?
- At the concentrations seen, what is the minimum size area to be considered as posing a risk to the environment or the surrounding population?
- Which areas must be treated in order to reduce the risk from exposure to an acceptable level?
- Which remedies can be applied at this site in order to clean up the soil?
- What is the volume of material that must be treated by the remedy?
- What is the source of the pollutants?
- Are there other sources from which the chemicals could have migrated onto the site from other outside areas?

The two components, RI and FS, are conducted as interdependent phases so that the data collection and assessment requirements of the RI complement and support the recommendations of the FS. The resulting report identifies possible remedial actions and makes specific recommendations.

A graphic illustration showing the relationship of the DQO process to the phased RI/FS approach is presented in U.S. EPA 1987b. Investigators must incorporate and apply DQO process requirements during the RI/FS scoping effort and after each RI/FS data collection activity.

Stage 2: Identify Data Uses and Needs

> Stage 2 results in the stipulation of the criteria for determining data adequacy. This stage involves specifying the level of data certainty sufficient to meet the objectives specified in Stage 1. In Stage 2, the needs and goals of the remedial investigation will be determined and all decisions to be based on information gathered during the RI specified. This stage also provides for the evaluation and selection of the sampling approaches and the analytical options and evaluation of the use of a multiple-option approach to effect a more timely or cost effective RI/FS (U.S. EPA, 1987a).

Identify Data Uses and Needs: Stage 2 starts after the evaluation of the existing data and the determination of how well the data fit the conceptual model that was designed. In rare cases after concluding Stage 1, there may be adequate data to make a decision without additional sampling. In most cases, the type, quantity, and quality of required data will be defined in Stage 2. One should then attempt to identify all of the expected uses of the data. The amount of detail will depend upon the level of the effort. In cases where soil sampling is only a minor part of a RI, the table would be very abbreviated. Where soil is the major component of exposure and a soil remedy is anticipated, the table could be extensive. The specification of the data types must be in adequate detail to address the objectives of the RI/FS.

A number of the new remedies such as fixation and soil cleaning that are being used require that components of the soil mass be segregated, screened, or processed in some manner during the remedy. It is impossible to determine the feasibility of implementing these remedies without physical data such as unit density, percent debris, percent moisture, etc. Also a number of non-standard chemical analyses may be required. These should be included in the Summary of Data Needs Table (Table 1) even though guidance for developing the DQOs (U.S. EPA, 1987a) calls for rather broad generic data uses.

TYPE OF ANALYSIS	ANALYSIS NEEDED FOR						
	Health & Safety	Site Character	Risk Assessment	Engineering Alternatives	Engineering Design	Contract Bidding	Concentration Patterns
CHEMICAL							
Metals	X	X	X	X	X		X
VOA	X	X	X	X	X		X
Semi-Volatiles	X	X	X	X	X		X
Other	X	X		X	X		
PHYSICAL							
Density				X	X	X	
Particle Size				X	X	X	
Material Composition	X		X	X	X	X	
MICROORGANISMS				X	X	X	
VAPORS	X	X	X				X

Table 1. Summary of Data Needs

Once the data needs and types have been identified, priorities must be set so that resources can be properly allocated to the sampling effort. This prioritization should be closely linked to the allowable level of Type I and Type II errors. The required data quality for the highest priority data use will control the planning and implementation of the sampling effort.

Since soil sampling is expensive, there is a tendency to attempt to acquire as much data as possible from a single study. It may be more cost effective to conduct the sampling effort in stages or increments (i.e., exploratory study followed by a more definitive effort). Data from an exploratory study can provide considerable guidance in identifying the types of samples needed, the analyses required, and the quality of data that can be expected for a particular sampling method. The example presented below shows how the results of the exploratory study can be used to identify the analytical needs of a study.

> Example: A transformer repair yard located in a small Florida town was sampled during an exploratory study. Soil samples were collected at three depths from twenty-five grid points over the site. Priority pollutant analyses were carried out on these samples. The only pollutants found were PCBs (reported as Aroclor

1260), trichlorobenzene and tetrachlorobenzene. No breakdown products of the PCBs or the chlorobenzenes were noted.

In this case, extensive use of priority pollutant analyses on any additional samples would be wasteful of resources. Analyses should be focused on the three chemicals identified. Verification of the findings of the exploratory study may be substantiated by submitting a limited number of new samples for priority pollutant analyses.

Evaluate Various Sampling and Analysis Options: Each component of the sampling process (i.e., type of sample and its associated analyses) must be identified and carefully evaluated to determine if the particular sample type and analysis will provide the necessary information to meet the use or data need.

An example would be an emergency response situation such as a spill site where exposure to the population is critical. The high concentrations found at these sites often can be detected by use of some Level I field instruments. Screening of the soil with a photoionization or a flame ionization detector, for example, could provide the necessary results for the immediate clean-up of the spill. Samples collected in and around the area identified by the Level I instruments may then be submitted to a field laboratory for Level II analysis. The areas identified as requiring clean-up could then be addressed for the emergency response action. A limited number of samples should be submitted for Level III or IV analysis. This phasing of the analysis can provide the rapid turnaround needed by the emergency situation and still provide the necessary high quality data needed for verification and possible litigation. For information concerning analytical levels appropriate for selected data uses, see U.S. EPA 1987a.

The use of various levels of analyses should be considered when allocating resources for the RI/FS. The cost of field methods (i.e., Level 1 and 2) time-to-data availability are usually considerably less than the cost and time of a Level IV analysis. By coordinating Levels I and II with the laboratory methods (i.e., Level III), a higher quality data set can be developed in less time and for less cost.

Acceptance Criteria: Westat (1988) outlines a process for determining when a particular environmental goal has been attained. This process can be used to assist in planning the system DQOs for a particular sampling effort. Figure 3, taken from Westat's report, outlines appropriate attainment objectives that should be developed by the DQO Team in conjunction with the decision makers. The decision criteria that are developed must take into consideration the action level and the acceptable risks associated with that level. The acceptable risk of a false negative, for example, is defined as the probability that an unremediated area will in fact exceed the action level.

There are few regulatory guidelines or standards for soils; therefore, it may be necessary to determine a reasonable action level for identifying potential areas in need of cleanup. Where multiple chemicals are present, one can prioritize chemicals according to their toxicity, mobility, persistence, and concentrations. An Agency publication (U.S. EPA, 1984) can be a resource in helping to select indicator chemicals.

It may not be possible to precisely define the cleanup area at a particular site at the outset. It may be possible to arrive at an estimated size for this area based on the conceptual model prior to the sampling. This can become the basis for defining the monitoring design approach.

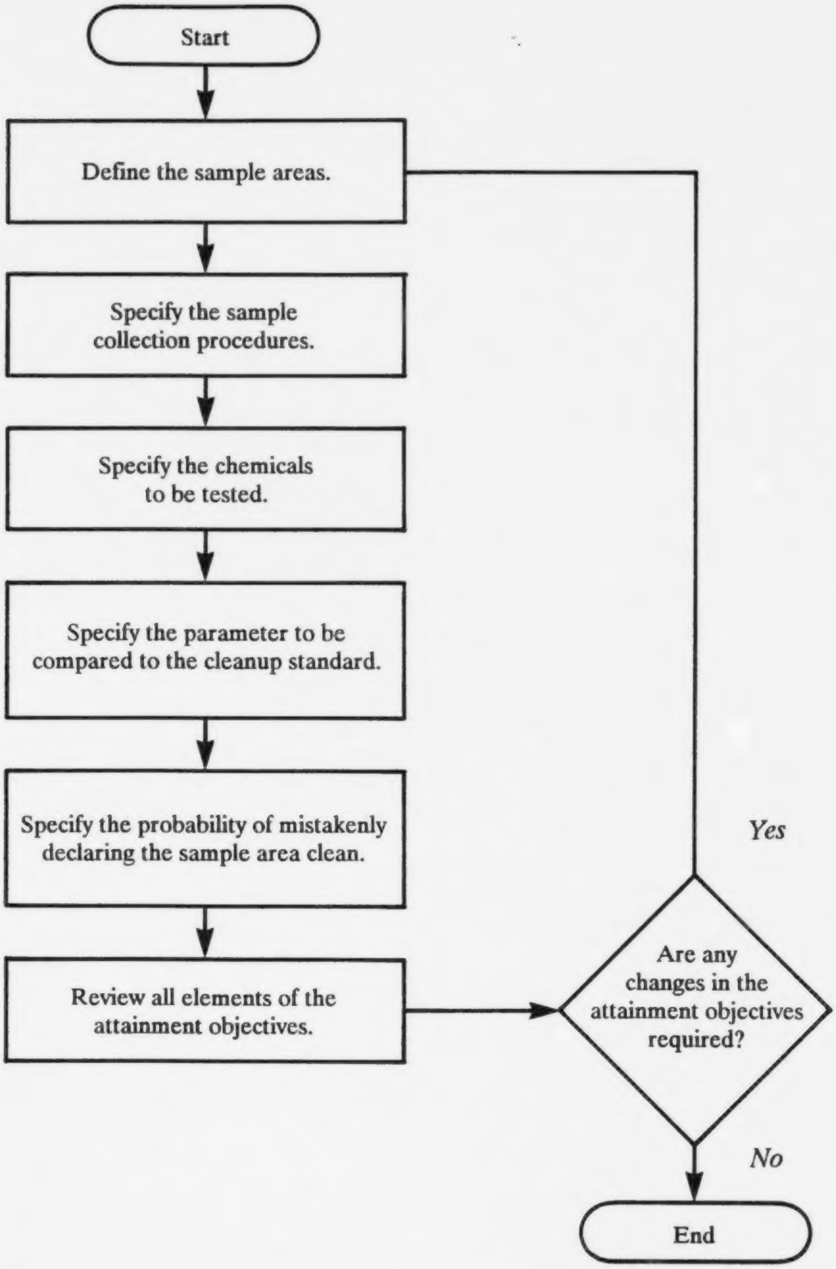
Start
Define the sample areas.
Specify the sample collection procedures.
Specify the chemicals to be tested.
Specify the parameter to be compared to the cleanup standard.
Specify the probability of mistakenly declaring the sample area clean.
Review all elements of the attainment objectives.
Are any changes in the attainment objectives required?
Yes
No
End

Figure 3. Steps in defining the attainment objectives.

Data Quantity and Quality Needs: Guidance for determining the number of samples that must be taken and the type of analyses that must be carried out is provided in Mason (1983) and in later chapters of this document. There must be a balancing between the number of samples and the resources available to meet the sampling/monitoring needs. A properly designed exploratory study will provide data needed for determining the final number of samples to be taken. In those cases where an exploratory study cannot be carried out, a phased sampling plan may be used. This allows the collection of an initial set of data that is used to design the next phase of collection. Addressing the questions presented below will be helpful in selecting sampling locations and numbers.

- Are there visible sources of pollutant on the surface of the soil?
- Is there soil erosion or recent cuts or fills on the site?
- What is the surface water flow pattern?
- Are there sensitive ecosystems or residences located down gradient from the site?
- Are there known hotspots on the site?
- Are there confining layers or porous layers in the soil horizon?

It helps to evaluate the form of the chemical pollutant, the media that transport it, the reactions that it may undergo, the routes that it may follow, and any sinks or other restraints that it may encounter as it moves from source to receptor. Those locations in the soil system where the pollutant is likely to be found should be sampled.

The required quality of the data can vary depending upon the use of the data. The evaluation of the existing data that was carried out in Stage 1 will indicate if the data are of adequate quality for the needs of the data users. The actual data quality that can be assigned to a particular data set can only be determined after the results are evaluated and interpreted. If the data do not meet the specified DQOs, additional data must be collected to bring the final data set up to the level of quality required to make the final decision.

Specify Precision, Accuracy, Representativeness, Completeness, Comparability, and Detection Limit Goals: The key phase of Stage 2 is the setting of the required levels of precision, accuracy, representativeness, completeness, and comparability along with the detection limits needed to meet the data quality objectives. All data uses do not require the same quality of data. "What is required, however, is that all data collected be of known and documented quality" (U.S. EPA, 1987a).

Detection Limits: Appropriate detection limits should be selected for the intended purposes of the sampling effort. Sampling to identify strata to be used in a later, more definitive sampling effort may be carried out with Level I or II techniques in the field. The minimum detection limit of these instruments may be quite high compared to laboratory methods. For example, measurements of background levels would not normally be done with these field instruments because of their high detection limits. Field instruments with a higher detection limit may be appropriate for use in areas that have high pollutant levels. If a 10 ppm clean-up level has been identified, it is not cost effective to use an analytical procedure with a parts-per-billion detection limit. A field gas chromatograph can provide the reliability for the clean-up work. A set of samples can be submitted to a laboratory to verify that the field analyses meet the desired standards.

Field audit samples with a low concentration can be used to determine the minimum detection limits for the measurement process being used. The variability data from the analyses of the field audit samples will provide a means for determining at what level analytical results should be reported without qualifiers.

The detection limit selected should reflect the risks associated with exposure to a particular pollutant. Detection limits should not be chosen *a priori* on the basis of the methods available for analysis, but should result from a review of the needs of the decision maker and the data users. A rule of thumb might be to use whenever possible a detection limit one order of magnitude lower than any level of concern. This allows values close to the concentration levels of concern to be reported and evaluated.

Precision: The precision required for a particular study will depend upon the difference between background levels and the action level. Measurements of chemicals that have a very low action level such as 2,3,7,8-tetrachlorodibenzodioxin (TCDD) will require much greater precision than would measurements of a chemical with an action level in the parts per million range. The amount of preparation that samples undergo prior to analysis will also greatly influence the precision of the measurement process. Soil samples that have been taken for metals analysis are often dried, sieved, and mixed, and then carefully subsampled. These subsamples provide a much more precise measure of the average concentration in the sample than would be expected from a sample that could not be prepared in the same manner. For example, samples collected for volatile organic analysis cannot be dried, ground, or mixed if they are to reflect the concentrations found in the soil.

Laboratory precision is only one part of the total precision of the measurement process leading from sample collection through data reporting. Selection of an acceptable precision level should not be based solely on what is attainable in the laboratory. Once the sample is submitted to the laboratory much of the sample-to-sample variation has already been introduced into the sample by activities in the field.

A key factor to remember when making a decision on the desired level of precision is that the selection should be made on the basis of risk of exposure if protection of public health and the environment is the principle matter of concern. The detection limits, the sampling methods, and the sample handling procedures must then be specified on the basis of the levels of pollutants judged harmful by the risk assessment. Where litigation is a key factor and costs are high, the choice of techniques for assuring adequate precision become important.

Normally precision is measured by the standard deviation of the data set; however, the range can also be used (Bauer, 1971). Replicate quality control samples are submitted from the field to provide a means of determining the precision of the measurement process. Two types of samples should be used for this purpose. Routine samples should be submitted as either splits or co-located samples. In addition to the routine samples, field audit samples also should be submitted on a regular basis.

Accuracy: Accuracy is controlled primarily by the laboratory and is reported as bias. Standards, spiked samples, referee samples, and field audit samples are all used to assess and control the accuracy of the results as well as the comparability of the results.

Representativeness: Representativeness is the degree to which the samples collected reflect the conditions at a particular site. For example, a sample of soil screened from a rubble pile does not represent the conditions at that site, but only provides a measure of a small fraction of material that happens to fall within the screened particle size range.

Sampling techniques and the monitoring design selected determine what is actually being measured. The rationale for selecting a particular technique or design (e.g., when, when, and how to sample) should be carefully defined and documented as to its applicability in defining site conditions. Samples that are biased toward hotspots should be identified. Sampling only suspected hotspots is often used in the initial stages of an investigation and insures that some potential problems will be identified quickly, but it generally provides only a limited indication of the magnitude of the total problem.

Completeness: Completeness is a measure of the amount of validated data that is obtained from a particular sampling scheme. It is calculated by dividing the number of validated data points by the total number of samples collected. The design of a particular sampling effort provides a minimum number of samples that is needed to yield a desired level of precision for the final results. The probabilities of false positive and false negative answers are specified at the outset. Obviously any loss from the required number of samples will impact the final results. The U.S. Department of Energy has set a completeness objective for the Environmental Survey Program at 90% for both field sampling and laboratory analyses (U.S. DOE, 1987).

The planning stages of any study must take into consideration the fact that not all samples will make it intact through the entire measurement process. Sample containers will be broken, instruments will fall out of control, data will be lost, sample tags will be lost, storage conditions will be violated, etc. There are many factors that can lead to a sample result being invalidated. This can be compensated for by oversampling or by using a phased sampling effort that allows areas where samples were lost to be resampled in subsequent phases. This latter approach insures that the desired number of samples will be collected.

The completeness goal must be realistic and must assure that adequate data will be available for meeting the objectives of the sampling program.

Comparability: The comparability objective provides the needed control over the total measurement processes to insure that different studies can be compared. Comparability provides a basis for comparing trends over time or space, for evaluating the relationship between sampling programs, and for insuring that phased sampling efforts produce data of a consistent quality. The quality control procedures used in the laboratory provide a portion of the control over comparability, but the field audit sample provides the best basis for insuring that data sets are comparable. A field audit sample from a previous study should be included in the first few batches of samples submitted from a new site. This allows comparisons to be made through the two sets of field audit samples.

When sampling is to occur over an extended period of time or when the investigator desires to compare several sites, it is necessary to insure that the samples be collected in a comparable manner, from comparable fractions of the soil mass, and with comparable methods. For example, one should not attempt to compare samples collected by coring with bucket auger samples.

Stage 3: Design of the Data Collection Program

> During Stage 3, the methods used to obtain and analyze data, as well as the quality and quantity of data required to achieve the objectives outlined in Stage 2 will be specified. This information is provided in documents such as the work plan or quality assurance project plan. Stage 3 results in the specification of the methods by which data of acceptable quality and quantity will be obtained (U.S. EPA, 1987a).

During Stage 2, specific guidelines have been developed for sample collection, chemical analyses, and data evaluation. The data quality objectives that were defined are used to design the procedures that will be used to acquire the quality of data that is needed to meet the demands of the decision maker and the data users. Stage 3 compiles information and merges it with the data quality objectives to arrive at a data collection program. The output of Stage 3 should be a set of well-defined and documented plans for acquiring the data and insuring that the quality of the data meets the DQOs. A U.S. EPA publication (U.S. EPA, 1987b) provides an example of DQOs for soil sampling that can be used as a guide.

Mason (1983) has prepared a manual for use in preparing soil sampling protocols. This document outlines sample design considerations and sampling techniques that can be used to prepare the documentation needed for a soil sampling effort.

A detailed protocol or work plan that spells out detailed instructions for every aspect of the soil sampling program should be prepared. Documentation should include instructions for acquisition and preparation of sampling equipment, sampling/monitoring design, health and safety, quality assurance, decontamination, disposal of wastes, sample and document control, analytical procedures and data validation and analysis. These aspects may be combined into one large document, but most likely it will be a series of documents addressing specific aspects of the entire measurement process.

To provide additional information and assistance to those responsible for designing and implementing sampling/monitoring programs, a Data Quality Objectives Development Process is presented in Appendix C. This process involves a four-stage interactive approach. The accompanying checklists and critical elements of a quality assurance plan are used by U.S. EPA Region 10 to address and identify site-specific Data Quality Objective requirements.

CHAPTER 4

QUALITY ASSURANCE PROJECT PLANS

The U.S. Environmental Protection Agency quality assurance policy requires that every environmental monitoring and measurement project must have a written and approved Quality Assurance Project Plan (QAPP) (U.S. EPA, 1980, 1986, 1987d). These plans are based on the data quality objectives that have been developed for the RI/FS. The QAPP becomes the primary instrument for directing the quality assurance effort for the project and for insuring that the DQOs are met. U.S. EPA policy requires that the QAPP contain the sixteen elements listed below.

1. Title page with provision for approval signatures.

2. Table of Contents. (This must include a serial listing of each of the 16 QAPP components.)

3. Project Description. (A general description of the project should be provided together with the intended end use of the acquired data. This should be closely tied to the objectives identified during the DQO stages.)

4. Project organization and responsibility. (List the key individuals, including the QA Officer, who are responsible for ensuring that the collection of valid measurement data and the routine assessment of measurement systems have met the DQOs.)

5. QA objectives for measurement data in terms of precision, accuracy, completeness, representativeness, and comparability. (For each major measurement parameter, list the DQOs for precision, accuracy, and completeness, detection limits, along with the probability of committing a Type I or Type II error that was used in defining the objectives. All measurements must be made so that the results are representative of the media and conditions being measured.

6. Sampling procedures. (For each major measurement parameter, including all pollutant measurement systems, provide a description of the sampling

procedures to be used. Any later changes to these procedures should be documented by attachment of approved amendments to these procedures.)

7. Sample custody. (Where samples may be needed for legal purposes, chain-of-custody procedures will be used. Most RI/FS data falls into this category. It will be necessary to define a detailed set of procedures to be followed by the field teams and the laboratories to insure that the chain-of-custody is followed.)

8. Calibration procedures and frequency. (Information should be provided on the calibration standards to be used and their sources.)

9. Analytical procedures. (Describe the analytical procedures to be used for each major measurement parameter along with the associated detection limits.)

10. Data analysis, validation, and reporting. (This section will include the principal criteria that will be used to validate data integrity during collection and reporting of data as well as methods used to treat outliers. This section should

also detail the procedures that will be used to insure that the DQOs have been met.)

11. Internal quality control checks. (Examples of items to be considered include replicates, spike samples, split samples, field audit samples, control charts, blanks, internal standards, span gases, quality control samples, surrogate samples, calibration standards and devices, and reagent checks.)

12. Performance and systems audits. (Each QAPP must describe the internal and external performance and systems audits that will be required to monitor the capability and performance of the total measurement system. The use of field audit samples should be outlined in this section.)

13. Preventive maintenance. (This section should include a schedule of important preventive maintenance tasks as well as inspection activities.)

14. Specific routine procedures used to assess data precision, accuracy, and completeness. (These procedures should include the equations used to calculate

precision, accuracy, and completeness, and the methods used to gather data for the precision and accuracy calculations. The types of control charts to be used along with the equations used to calculate control limits should also be included in this section.)

15. Corrective action. (This section must include the predetermined limits for data acceptability beyond which corrective action is required as well as specific procedures for corrective action.)

16. Quality assurance reports to management. (These reports should include periodic assessment of measurement data accuracy, precision, and completeness as well as an identification of significant QA problems and recommended solutions. The interim reports should specifically outline any problems that will cause failure to meet the DQOs. The final report should address how well the data quality objectives for the study have been achieved, along with any reasons for failure to meet these objectives.)

Since the original design of the QAPP, the use of data quality objectives has been implemented. The data quality objectives define the standards that must be met in order to insure that the quality of the data meets the needs of both the decision makers and the data users. Properly implemented, these objectives become a powerful tool in the overall RI/FS process. The QAPP becomes the roadmap for confirming achievement of the objectives outlined during the DQO process. To be maximally effective, QAPPs should be designed in such a way that out-of-control situations are detected at the earliest possible time so that corrective actions may be taken quickly to avoid wasting valuable resources.

CHAPTER 5

THE CONCEPT OF SUPPORT

The better known texts on sampling theory and methods (e.g., Hansen *et al.*, 1953; Cochran, 1977) deal primarily with the sampling of people, housing units, or businesses where sampling units are discrete and well defined. In the sampling of a continuous medium such as soil, it is necessary to emphasize the definition of a sampling unit. In addition to a specified location, each soil sampling unit has a certain three-dimensional volume, shape, and orientation. These latter three characteristics when taken together are called the **support** of the sample. The concept of support is somewhat analogous to the concept of a pedon used in soil classification work (Soil Survey Staff, 1975).

The choice of support will affect the characteristics of the distribution of the pollutant concentrations of the population of possible sampling units in the region being sampled. For example, if the sampling unit is a soil core and pollutant concentration decreases with depth, then the longer the core, the smaller is the mean concentration of pollutant in the sampling unit. However, changes in support not only change the means of the distributions, they also change the variances of concentrations and the correlations of concentrations between sampling units. Large variances of pollutant concentrations between sampling units often necessitate taking large numbers of samples or cause a compromise that results in larger than desired variances of sample estimates. In many cases, a change in the support of the sampling unit will substantially reduce the variance between sampling units and thereby reduce the variances of sample estimates.

Since one of the objectives of QA is to estimate the precision of measurements and sample estimates, it is essential that the support of all sampling units within a study be the same. However, it is possible to change support between an exploratory study and the definitive study and still be able to use the data from both studies in making estimates. This is permissible when the changes in support have not altered the expected values of the concentrations in the sampling units, and the data from the two studies are weighted to reflect the differences in variances of the measurements.

Soil is a very heterogeneous material. Samples with very small support volume (say 1 mm^3) may vary from zero to very high concentrations, regardless of the concentrations in samples of larger volume from the same location. With no prior information, it is difficult to choose an appropriate support. With information as to the spatial continuity of the pollutant concentration in the soil, one can follow a systematic procedure to determine the appropriate support for a soil sample. (See Starks, 1986.) In a later paragraph, it is shown how quality assurance samples can also give information on the appropriateness of the chosen support.

Often one finds action levels expressed as a certain number of parts per million or parts per billion, but for soils, such statements of action level have little meaning. As mentioned above, for sampling units with very small volumes, it is almost certain that some samples will be above action level. For example, suppose that all the pollutant were concentrated in particles of size 1 mm^3 and that each of these particles has 10 times the action level concentration. If these particles were uniformly distributed in the top ten cm of the soil in the area of interest, and the total volume of all such particles were only 0.1 percent of the total volume of the soil in the top ten cm, one would conclude that no remedial action is necessary. However, if the particles were all concentrated in the soil so that they formed a "hot spot" of perhaps the top ten cm of one acre in a 1,000-acre region, it would be important to locate the hot spot so that remedial action could be taken. Hence, it is essential that the action level be defined as a concentration over a particular support and location relative to the ground surface.

In this definition of action levels, the support will be referred to as the **action support**. For example, one might state as an action level that if there is a soil volume that has a concentration of pollutant X exceeding 10 ppm in the top ten cm over a square area of at least 100 m^2, then remedial action should be taken on that volume. In this example, the action support is the top 10 cm over a square area of 100 m^2.

At the Palmerton, Pennsylvania, Superfund site, cadmium was one of the soil pollutants of interest (Starks *et al.*, 1987). The support of soil samples taken in an exploratory study was a set of four (2 cm diameter) cores taken to a depth of 15 cm with one core from each of the four cardinal compass points on a 6 m diameter circle. The four cores were composited at all but 10 of the 211 sample locations. At ten locations, measurements were obtained from each of the four cores. At another 10 sample locations, the sampling team took a second (duplicate) sample of four composited cores within 0.5 m of each of the original four cores. (See Table 2.)

The variance between measurements on the duplicate pairs was 31 percent of the total variance between samples (after correcting for changes in expected values over locations). The variation between measurements on individual cores was found to be quite large. (See Table

3.) Other QA sample results indicated that the large variances found between duplicates and individual core measurements were not caused by subsampling or chemical analysis errors.

To reduce the large component of total variance caused by the placement of the sample cores, it was decided that nine cores should be taken at each sampling location in the second (definitive) study. The nine cores came from four points at the cardinal compass points of a 6 m circle, from the four minor compass points of a concentric 4.25 m circle, and one point at the center of the circles. This increase in the support of the samples, plus the increased experience of the sampling teams, brought the variance between duplicates down to less than 10 percent of the total variance in the definitive study.

TABLE 2. RESULTS FROM DUPLICATE SAMPLES AT THE PALMERTON SITE

(mg/kg)

POINT	CADMIUM		D[a]	L[b]
AK26	8.85	7.35	1.50	0.186
AO30	22.30	8.46	13.84	0.970
BP30	86.30	63.10	23.20	0.313
BQ33	58.20	40.30	17.90	0.368
BQ34	100.4	77.10	23.30	0.264
BT32	39.50	37.90	2.40	0.041
BT46	172.0	144.0	28.00	0.178
BY29	43.70	34.20	9.50	0.245
DL78	2.76	2.71	0.05	0.018
DP34	68.10	61.70	6.40	0.098
Median:			11.67	0.216

$s^2 = \Sigma L_i^2/20 = 0.0691$

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[a] D = absolute pair difference

[b] L = absolute pair difference of log-transformed data

TABLE 3. RESULTS FROM INDIVIDUAL CORES AT THE PALMERTON NPL SITE
(Cadmium, mg/kg)

SAMPLE	CORES					
POINT	S	W	N	E	RANGE	V*
BO29	30.7	5.0	66.3	29.0	61.3	1.195
BR33	56.7	66.8	25.8	104.3	78.5	0.339
BR38	104.0	147.0	124.0	100.6	46.4	0.031
BS33	42.9	21.2	30.5	45.5	24.3	0.124
BU33	29.4	24.1	31.0	48.6	24.5	0.877
BU38	64.0	33.1	82.3	127.0	93.9	0.316
BX30	56.3	35.9	46.2	111.0	75.1	0.234
CF46	51.5	97.1	46.7	79.0	50.4	0.121
CI34	112.0	167.0	158.0	196.0	84.0	0.055
CJ50	0.75	47.4	115.0	52.0	114.25	5.159

* Sample variances on ln(Cd) over 4 cores at each site.
Pooled sample variance based on first nine sample points: 0.3659.
Pooled sample variance based on all ten sample points: 0.8453.

CHAPTER 6

EXPLORATORY STUDY

Once objectives which involve the need for soil sampling have been defined, the next step is to develop a total study protocol including DQOs as well as an appropriate QA/QC program. Answers to the following questions should be available or estimates made in order to develop the protocols.

- What are the probable sources of the pollutants of concern?
- How have the emissions from these sources varied in the past compared to their present levels?
- What are the important transport routes which contribute to soil contamination?
- What is the geographic extent of the contamination?

- What average concentrations of the pollutants exist at different locations, and how do these vary as a function of space and time?
- Do localized areas of high concentrations exist, and if so, where are they and what are the concentrations?
- Is it possible to stratify the sampling region to reduce the spatial variations within strata?
- What are the soil characteristics, hydrological features, meteorological or climatic factors, land use patterns, and agricultural practices affecting the transport and distribution of the pollutants of concern in the soil?
- What is an appropriate background or control region to use for the study?
- What are the acceptable levels of precision, minimum relative levels of detectability, and probabilities of both Type I and Type II errors for this study?

If answers to all of these questions are not available, an exploratory study (this also can be called a pilot study or a preliminary study) should be carried out. To be designed after a site visit, this study should address the components of a conceptual dispersion model. Clearly not all the above questions can be answered in detail by a single exploratory study; however, as many as possible should be attempted.

The authors recommend developing the conceptual model after a compilation of literature and all existing data have been completed in Stage 1 of the DQO process. Much of the information pertinent to the above questions may already be available in the published literature, in the files of governmental agencies or industrial corporations, in ongoing or completed research at local universities, or in the knowledge of local citizens. A carefully planned and organized effort should be mounted to accumulate what relevant information is available. Only after this information has been collected, collated, and evaluated should any field measurements be made. It is good policy to adhere to a reasonable fixed period of time for the collection and analysis of available information, otherwise this process could drag on interminably. Only at the end of the fixed time period, and based on whatever information is available at that time, should the design and implementation of the field measurements portion of the exploratory study be undertaken.

In those cases where there is not enough data available for designing the soil sampling study, an exploratory study becomes an essential element of the planning process. Properly designed, the exploratory study is simply phase one of a multiphased sampling effort.

The primary intent of the exploratory study is to determine if the site poses a potentially unacceptable risk to the local human population or the environment. The study also assembles and collects data needed to prepare revised Data Quality Objectives, work plans, QAPPs, and sampling protocols.

The designs used to acquire the data during the exploratory study should have a statistical basis that provides some measure of the precision that can be expected for the particular soil-pollutant combinations that exist on the site. The authors recommend that a coarse grid pattern be used in order to provide some information on the distribution of the chemicals over the site. The investigators may decide that a combination of judgmental sampling and systematic sampling may provide more useful information for a particular situation. Those samples that are selected on a judgmental basis are biased toward finding pollution. Because of this bias, the samples should be identified in such a way that the statistician can take the bias into consideration when providing a statistical analysis of the data. One possible alternate approach is to stratify the study area on judgmental evidence and then take random samples within each stratified area.

Aerial photographs of the site and its vicinity along with appropriate maps should be assembled before the exploratory study. This provides a basis for evaluating terrain, surface water flow, erosional patterns, land use patterns, and other similar factors that may influence the distribution and dispersal of the pollutants over the site. Careful attention should be paid to the identification of the primary sources of the pollution and to the primary routes of migration. These should be noted on the maps and incorporated into the conceptual model of the site.

The conceptual model of the site should be used as the basis for designing the sampling effort. Those areas deemed to be of primary concern should be sampled in such a manner that the model can in fact be verified or amended as needed. This involves incorporating into the sampling design adequate replication on several scales to determine the expected spatial variation over the site. Assumptions may have to be made about the expected variation that one would expect to encounter based upon similar situations at other sites. These would then be adjusted in the final sampling effort to incorporate the information gained in the exploratory study or preliminary site investigation. The Palmerton NPL Site example discussed below is an excellent example of the use of the preliminary study to guide the final sample design. The short-range variation (i.e., variation between samples 0.5 m apart) among results from neighboring grid points was considered to be too large based on the preliminary study. This was corrected by altering grid spacing to effectively reduce the influence of the short-range variation on the final results.

Guidance for the confidence level ($1-\alpha$) and the power ($1-\beta$) of the data set that should be used for the exploratory study are given in Chapter 7 along with the relative increase over background that should be used in designing any random statistical sampling plans. This chapter also recommends the number of quality assurance and quality control samples that should be taken during the exploratory study.

EXAMPLE OF AN EXPLORATORY STUDY

The Palmerton NPL Site is used as an example of the use of an exploratory study to develop the guidance that is needed to develop the main phases of a definitive soil sampling program. In the narrative that follows the reasoning for the sample design, reasoning for transformation of data, and the types of quality of assurance data are discussed. (For more details than can be presented here about the study, the reader is referred to Starks *et al.*, 1986; Starks *et al.*, 1987; and U.S. EPA, 1989.)

The purpose of the entire study was to determine the spatial distribution of certain metals (principal interest was in the concentrations of cadmium and lead) in the soil in residential and farming regions near two zinc smelters. The two smelters are located on the southwest and the southeast edges of Palmerton, Pennsylvania. (See Figure 4.) This information was needed to plan remediation procedures for the area. The site RPM requested a geostatistical analysis of the data to obtain kriging estimates of the spatial distribution of the metals in the soil. The purpose of the exploratory study was to estimate the extent and the spatial structure of soil contamination by cadmium, copper, lead, and zinc.

Sample Design

As stated above, the principal objective of the exploratory study was to obtain information for estimating spatial structure and extent of the pollution. Estimation of spatial structure requires information on how concentrations vary with location and how differences in concentrations vary with distances between sampling units. A square grid of sampling locations

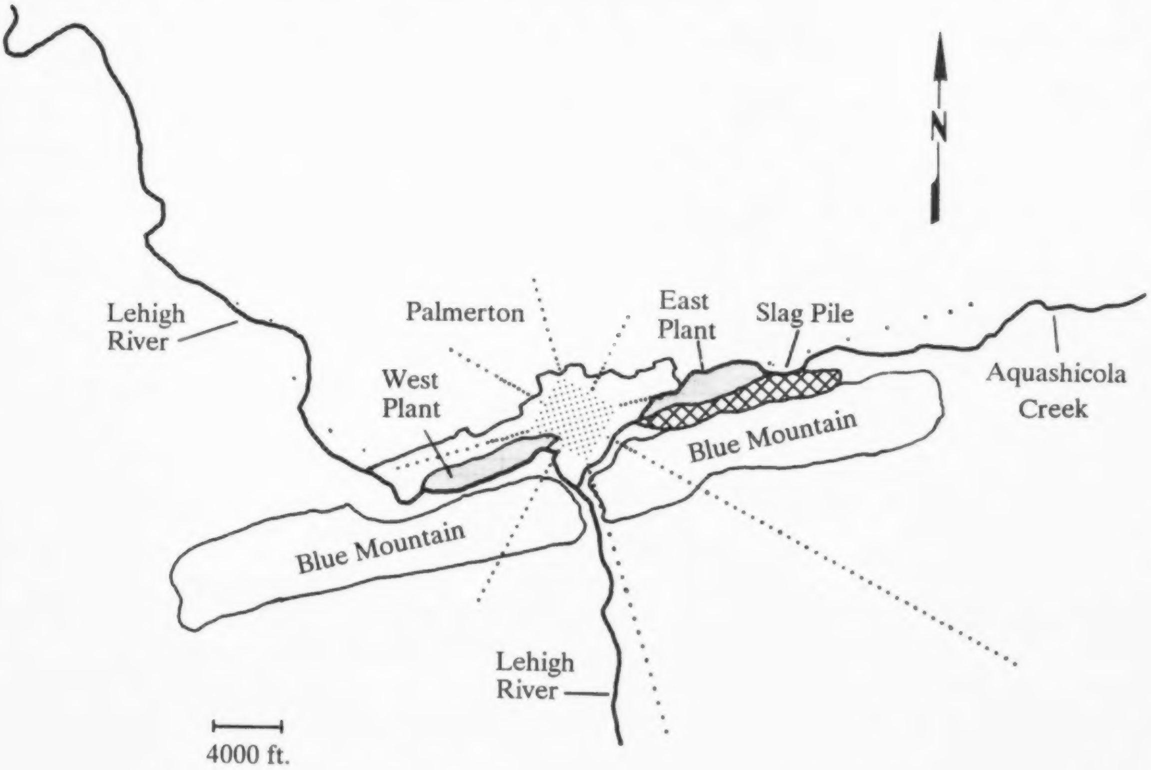
N
Lehigh River
Palmerton
East Plant
Slag Pile
West Plant
Aquashicola Creek
Blue Mountain
Blue Mountain
Lehigh River
4000 ft.

Figure 4. Palmerton Exploratory Sampling Design.

is good for this purpose in that each interior point can be associated with several other points a given multiple of the unit grid distance from it. The unit grid distance is of importance in that in that if it is too large, no spatial correlation will be detected; and if it is too small, more information than necessary will be obtained, and either a very small area will be sampled or a very large number of samples will be required.

In planning this study, the designers used results from a study of lead pollution in soil in Dallas, Texas, which indicated a range of influence (a distance beyond which there was no spatial correlation) of about 1,200 feet. Based on this information, a unit grid spacing of 400 feet was selected. This provided pairs of points at four distances (400, 800, $400\sqrt{2}$ and $800\sqrt{2}$ feet) at which positive spatial covariances might be expected for use in estimating the covariance function. To obtain a reasonable number of such pairs, a rotated square grid of 85 sample points was formed over a diamond-shaped area. (See Figure 4.) To obtain information on the extent of metal pollution in the soil, additional sample points were selected along eight transects originating at the center of the diamond and extending through its sides and vertices.

This grid of sample points was centered in the Town of Palmerton and oriented in such a way that three of the transects conformed to the valley system in which the town lies. One transect was bent to follow the Lehigh River. These transects also followed the principal windrose directions. The lengths of the transects were based on windrose data and land use data. The number of samples to be collected and analyzed had to be kept small, so that there would be resources to carry out the definitive study. Thus, spacing between points along the transect was generally greater than within the square grid. By centering the design in Palmerton, it was unnecessary to wait for the definitive study to obtain kriging estimates of metal concentrations for the area where most of the people in the study region lived.

Data Transformations

The need to transform data arises from the need to stabilize variances. If variance changes from location to location, there is no one variance to estimate. In addition, the kriging estimation process in geostatistics is variance-based and, therefore, requires a stable variance. The data in Tables 2 and 3 indicate that the variability in concentration measurements between duplicates and between cores from the same site tends to increase as the average concentration increases, and the same occurs between splits from the same sample. These phenomena were also observed in the concentration measurements for the other three metals. This indicates the need for a data transformation. Several methods for making a choice of transformation are given by Box and Cox (1964) and Hoaglin *et al.* (1983). However, estimates of proper transformations based on the small amount of data in the above mentioned tables are rather unreliable. In this case, it was known that the logarithmic transformation had been applied to these types of measurements in the past, and it was found to do a good job of stabilizing variance on a larger set of duplicate lead measurements from the Dallas study. Hence, the simple logarithmic transformation ($Y = \ln X$) was employed.

Graphical plots of means versus standard deviations for the log-transformed metal concentrations data showed no indication of a relationship between the two, and the variances of duplicates for the four metals were very close to being the same, even though the mean concentrations were quite different. This is as it should be, since the process of accumulation in the soil should be the same for all of the metals.. Similar inter-metal confirmations of the logarithmic transformation were found in the data from split samples.

Quality Assurance Data

Several types of quality assurance data were collected in the Palmerton exploratory study. These included duplicates, splits, and individual cores. In addition, decontamination blanks and QC samples were analyzed. A decontamination blank was collected at one sample location out of every 20. This blank was obtained by bringing the sample corer into contact with distilled deionized (DDI) (ASTM Type II quality) water (final rinse) prior to its use in taking the soil sample. The blanks were prepared in the field at the sample locations, then shipped with the field samples through the sample bank to the soil chemistry laboratory to determine whether the sample collection instruments were contaminated prior to taking a soil sample. Additional decontamination blanks were prepared in the sample bank by bringing DDI water into contact with the soil sieve and mixing equipment. One such blank was prepared after 40 samples were passed through the equipment to determine whether it was being properly cleaned between samples. Four QC samples consisting of DDI water with known quantities of Cd, Cu, Pb, and Zn were prepared and sent through the sample bank to the laboratory for accuracy checks.

If anything, this exploratory study was a bit short on QC samples. At least 20 duplicate samples should have been taken to allow better estimation of both the measurement error variance and the appropriate data transformation. In addition, no field audit samples (performance evaluation soils) were employed to check the precision and accuracy of the methods by using a soil matrix.

CHAPTER 7

GUIDANCE FOR SPECIFIC SOIL SAMPLING PROGRAMS

Chapter 2 discussed some of the functional objectives for soil sampling. The material that follows presents guidance for determining the confidence level, the power, and the detectable relative difference between different data sets that should be anticipated for different types of soil sampling programs. Most of the information relates to the provisions and intent of RCRA and CERCLA. Operational situations in which soil sampling may be involved include:

- background monitoring,
- preliminary site investigation,
- emergency cleanup operations,
- planned removal operations,
- remedial response operations,
- monitoring, and

- research or technology transfer studies.

With the possible exception of research or technology transfer studies, all of the operational situations listed have a potential for litigation. For this reason, a statistical experimental design incorporating appropriate QA/QC measures including National Enforcement Investigation Center (NEIC) "chain-of-custody" procedures should be incorporated into the overall sampling program. The total QA/QC plan should be designed to insure that the data quality objectives are met.

Background Sampling

Sampling to determine the background levels of the various chemicals found in the environment should be carried out as part of any routine sampling programs. Background levels are usually found in those areas where the levels are below the minimum detection limits. However, certain of the trace metals may be present at levels that are detectable and still be background levels. In order to determine if a specific area is contaminated above background, it may be necessary to carry out studies with this specific objective in mind.

Background areas should be areas outside industrial complexes that may be contributing to the overall pollution burden and should be upwind and upstream from them. These areas should be in similar topographical settings and have the same or very similar soil types. The parent material for the soil should be the same if at all possible. When the same soil type cannot be found, care should be taken to insure that the amount of organic matter and clay is similar in the soils chosen for the background areas.

These factors become especially important when chemicals normally found in the soil are the pollutant of concern. Sensitive analytical methods with low detection limits will detect many of the metals in the soil. When these are detected in the soil near a site, care must be taken in interpreting this as an indication of pollution.

Preliminary Site Investigation

The purpose of a preliminary site investigation or exploratory study is to provide information about a specific site that can be used in making initial management decisions and, should further work be necessary, for designing a more detailed and comprehensive sampling investigation. Since the data collected during the preliminary study will be used to make important decisions about the site, it is essential that the reliability of the data be demonstrated through incorporation and implementation of adequate QA/QC. For example, the preliminary results may indicate that an emergency response should be initiated. Making an erroneous decision based upon data of unknown quality could lead to serious and costly consequences.

Emergency Cleanup Operations

The purpose of an emergency cleanup operation is to remove enough of the pollutants as quickly as possible to achieve a level that is considered an acceptable risk to human health or the environment. The principal role of the QA/QC plan in this situation is to provide a reliable demonstration that cleanup operations have been adequate. An emergency cleanup operation often leads to a requirement for either a planned removal or a remedial response operation. Thus, any soil sampling undertaken during the emergency phase should have adequate QA/QC measures incorporated into the study to ensure that the resulting data may be used as a foundation for any subsequent investigations.

Planned Removal and Remedial Response Operations

The purpose of planned removal or remedial response operations (they differ principally with regard to time scale) is to provide a more permanent solution to the problem. These operations and the associated RI/FS may involve extensive sampling and data analysis programs. Adequate QA/QC measures are essential, since litigation to recover the costs of the operations is a probable sequel. Consequently, all data collected may be closely examined in court.

Monitoring

Monitoring, or sequential measurements over time, may take place before, during, or after any of the operational situations listed above. Whatever trends are measured must be demonstrated to be reliable in order to serve as a basis for making decisions that hold up to challenges.

Research or Technology Transfer Studies

The purposes of research or technology transfer studies vary widely. In any event, the incorporation of adequate QA/QC plans into these studies is mandatory in order for the results of the studies to withstand the normal peer review processes required for publication and/or application of the findings.

In summary, an adequate QA/QC plan should be part of any soil sampling program relevant to any of the operational situations listed. The only remaining question pertains to the definition of the word "adequate." The sections that follow discuss the above study types in more detail.

OBJECTIVES FOR BACKGROUND MONITORING

Generally, the design of soil monitoring programs requires that the levels of defined hazardous or potentially hazardous substances and their spatial and temporal trends be measured for some specific purpose. Often it is critical not only to quantify levels and trends, but also to link the existing levels to sources. This is necessary to enable adequate control actions to be taken whenever a situation that is hazardous to human health, welfare, or the environment is identified. The situation often is complicated by the fact that multiple sources contribute to the measured levels. The situation is further complicated by the presence of pollutants of recent origin mixed with pollutants of past origin. This mixing becomes especially important when the investigator attempts to trace the migration from source to receptor and also in predicting future levels after various proposed control measures are implemented. Identification of spatial and temporal trends, along with linkage of observed measurements to sources, requires that adequate background, reference, or control samples be taken.

In the absence of such background samples, interpretation of the resulting data may become extremely difficult, if not impossible. The burden of proof that background samples are not necessary for a particular soil monitoring study rests with the principal investigator. In the absence of such proof, a prudent investigator will ensure that an adequate number of background samples be included in the monitoring study design.

Since measured levels in presumably higher concentration areas will be compared to background levels, QA/QC procedures are just as critical for the background measurements as they are for the study area measurements. Thus, for background sampling, a QA/QC procedural umbrella must cover the selection of appropriate geographical areas; the selection of sampling sites within the geographical areas; sampling, sample storage and/or preparation; sample analysis, data reduction, and interpretation of study results.

Under most circumstances, background data will not be available for a given monitoring location. These data must be acquired either before or during the exploratory or preliminary investigation phase. The intensity of the background sampling that is undertaken depends upon the pollutants being measured, the soil characteristics and variability, the levels of pollutant likely to be found in the study area, and the purpose of the study.

SPECIFIC OBJECTIVES FOR MONITORING IN SUPPORT OF CERCLA AND RCRA

The principal sampling media now being measured to carry out the provisions and intent of CERCLA and RCRA are soil and groundwater. Hazardous constituents from a hazardous waste facility may enter soils through transport of the constituents from the waste site to soils via organic solvent, surface water, or groundwater flow. Air transport followed by dry precipitation, rainout, or washout will generally be less important than other transport routes.

Suppose a situation exists in which hazardous waste constituents have been leaving a site for a relatively long period of time and nearby soils have built up considerable levels of pollutants. Further, suppose that the soils now constitute a source of the hazardous constituents. At this time, removal of the hazardous wastes from their original disposal site may still leave a significant unsolved problem in the form of the contaminated soils, which may cause human exposure through skin contact or through ingestion or inhalation of soil particles. Also human foods, contaminated directly or indirectly through contact with soils, may be unfit for human consumption. Furthermore, as the hazardous constituents move through different trophic levels, substantial biomagnification of contaminants may take place, thereby increasing the risk to humans consuming foods from higher trophic levels. Thus, it is conceivable that situations may exist in which concentrations of hazardous constituents in soils may represent a major risk to human health or the environment. To identify such situations, data from soil sampling is an important link in the chain of required evidence.

The specific QA/QC precision and confidence level objectives for any sampling study are controlled in part by the goals of the particular study. Three situations where soil sampling would probably be undertaken are:

- hazardous materials investigations for areas such as abandoned landfills or chemical spills,
- monitoring studies, and
- technology transfer.

The data flow that can occur in each of these situations is outlined in Figures 5, 6, and 7. The data generated in each category can provide input into the development of plans and specifications for the other situations. Data that have been subjected to a good QA/QC plan can be relied upon as a resource for the development of new data.

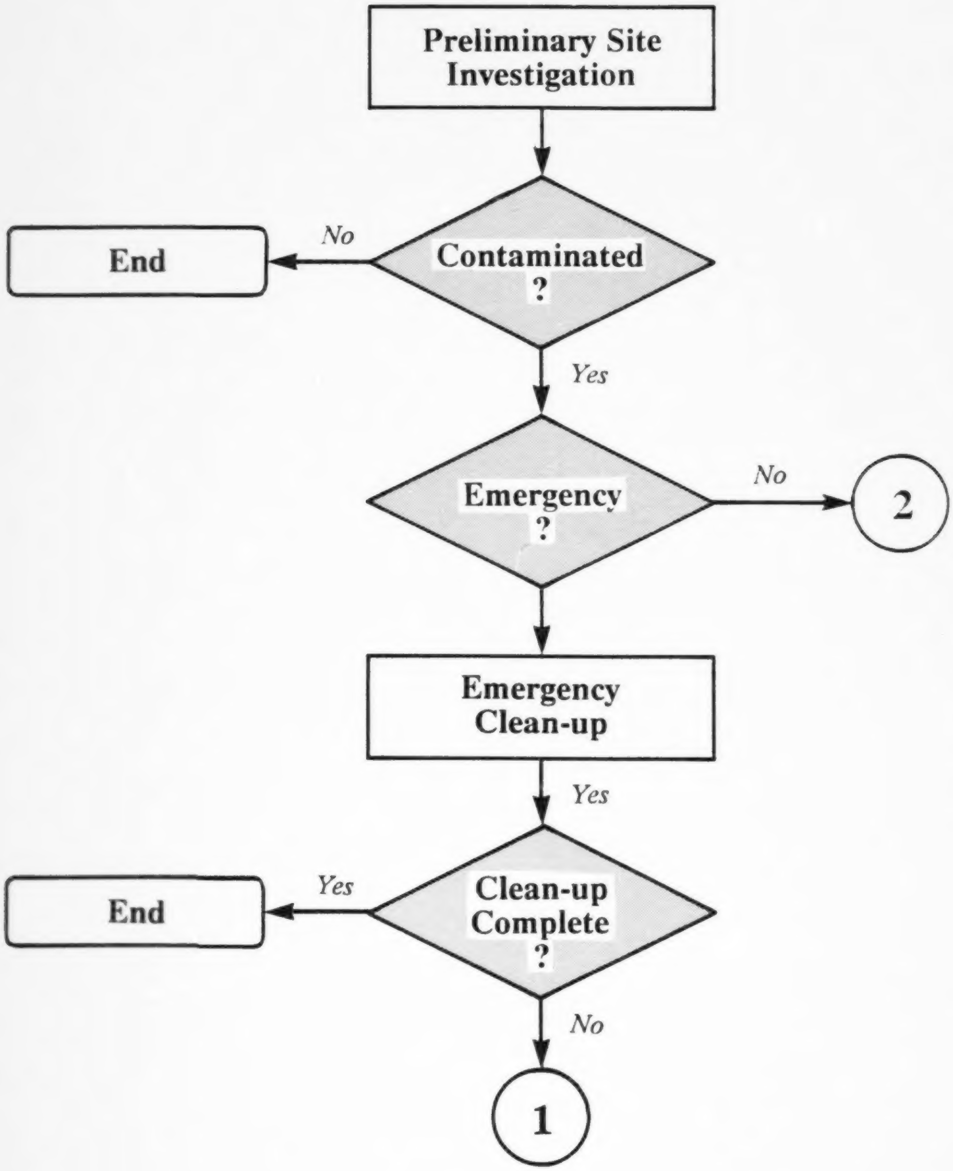
Preliminary Site Investigation
No
End
Contaminated ?
Yes
Emergency ?
No
2
Emergency Clean-up
Yes
Yes
End
Clean-up Complete ?
No
1

Figure 5. Data acquisition flow for hazardous materials.

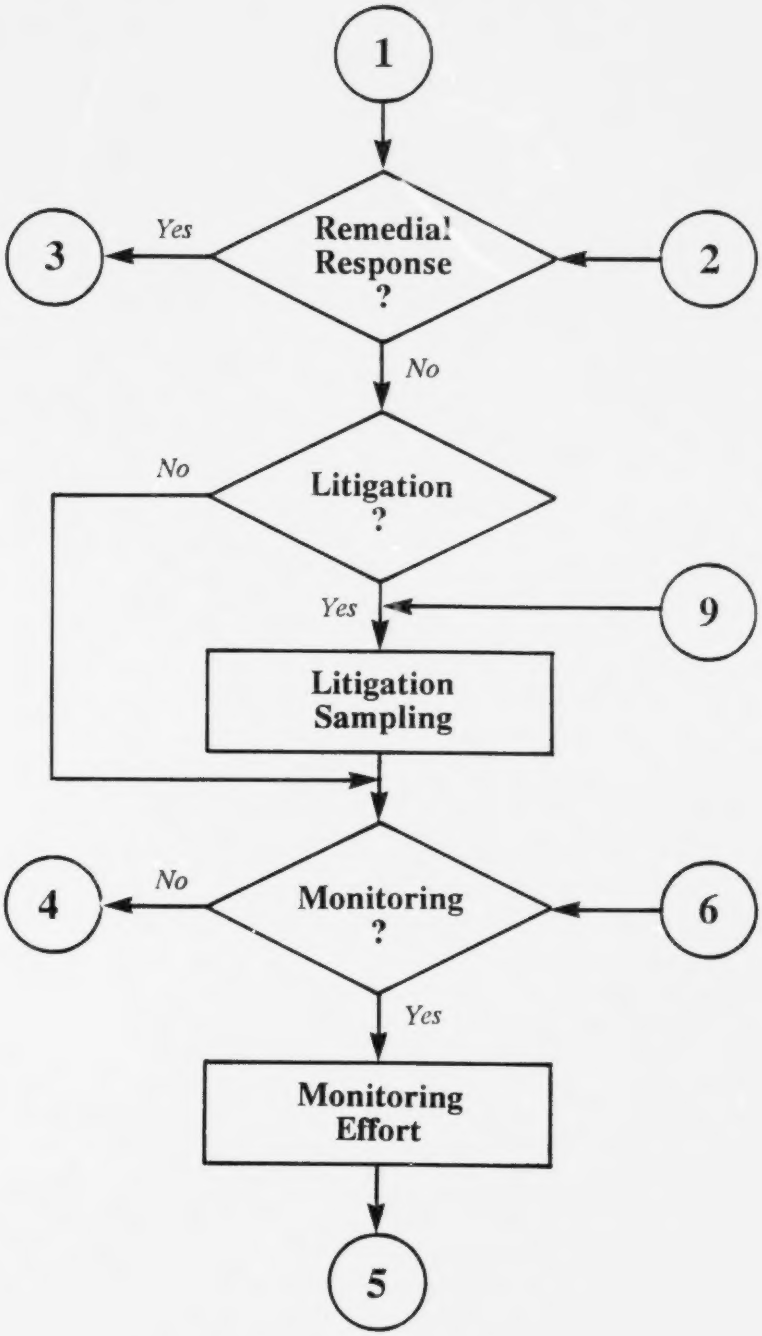
1
Yes
Remedial Response ?
3
2
No
No
Litigation ?
Yes
9
Litigation Sampling
No
Monitoring ?
4
6
Yes
Monitoring Effort
5

Figure 5. (Continued)

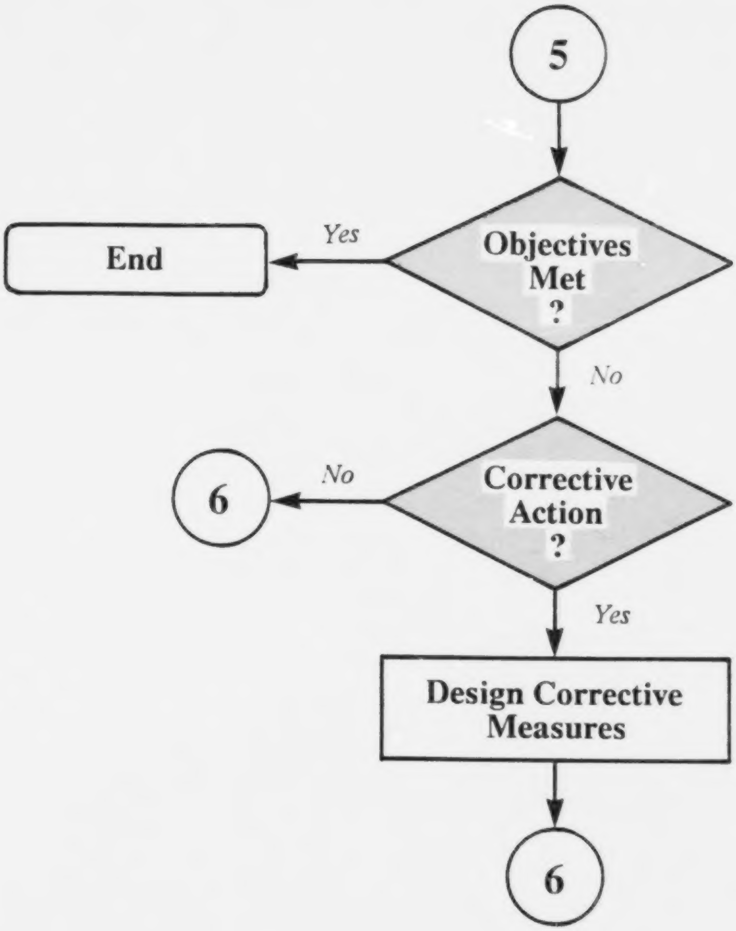
5
Objectives Met ?
Yes
End
No
Corrective Action ?
No
6
Yes
Design Corrective Measures
6

Figure 5. (Continued)

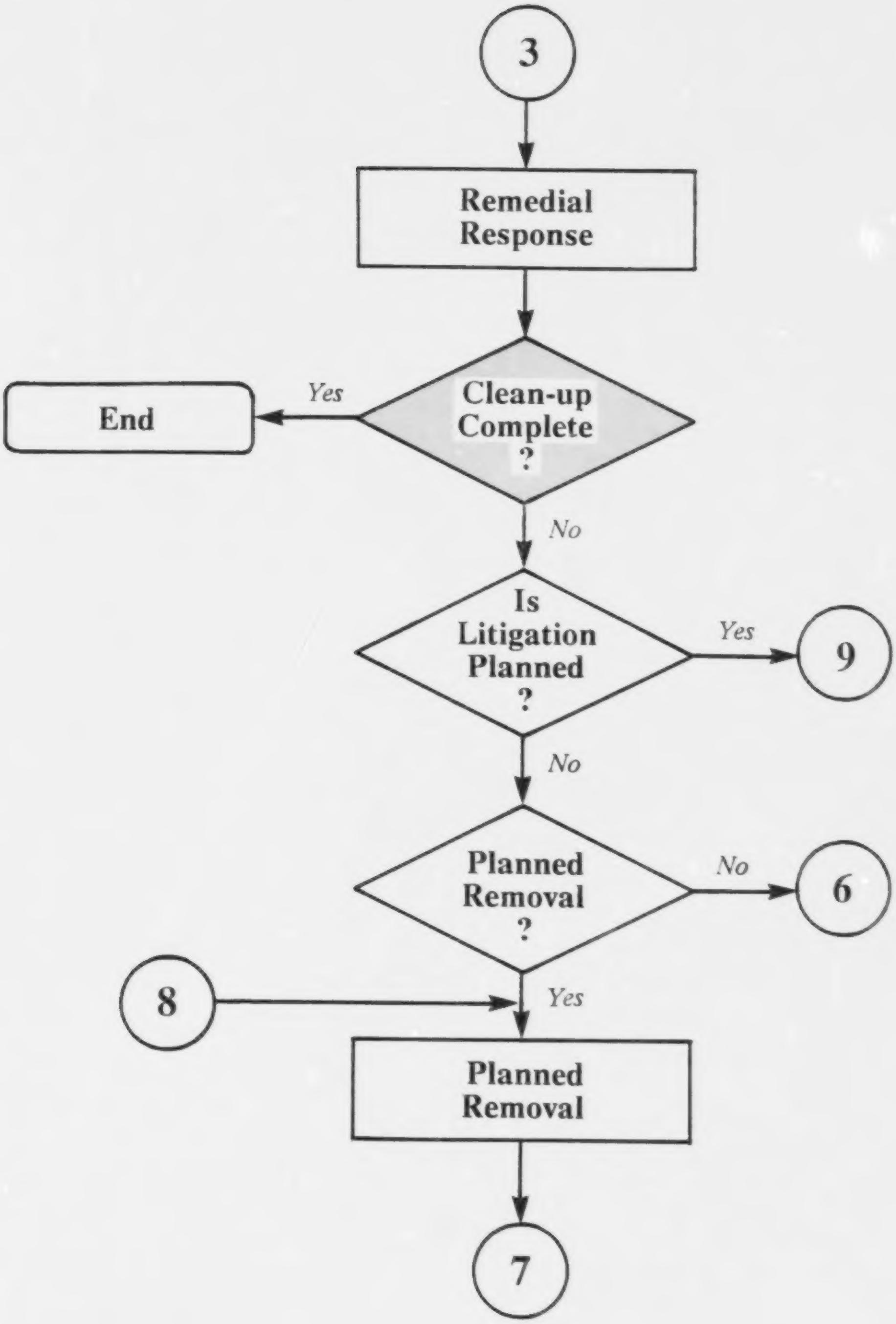
3
Remedial Response
Clean-up Complete ?
Yes
End
No
Is Litigation Planned ?
Yes
9
No
Planned Removal ?
No
6
8
Yes
Planned Removal
7

Figure 5. (Continued)

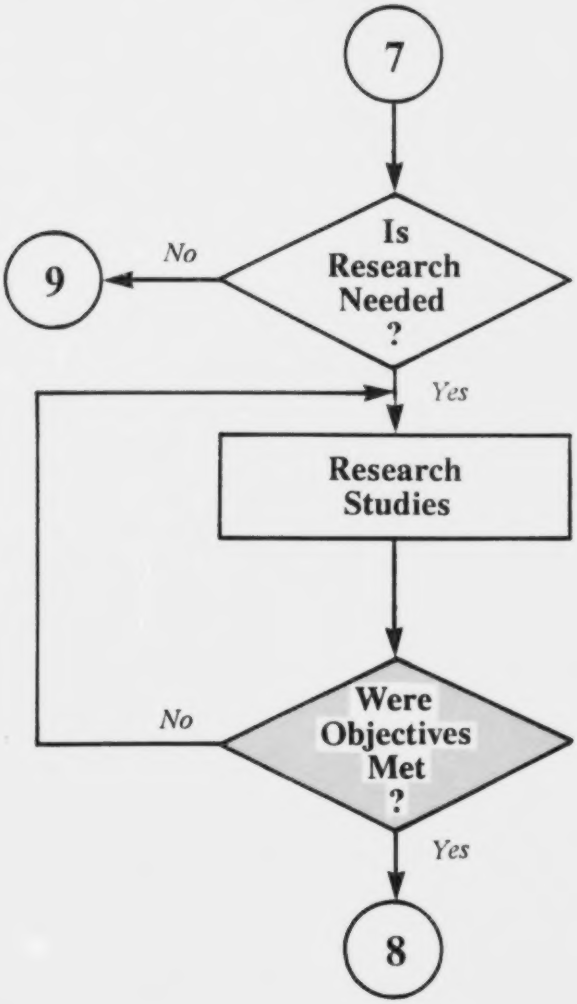
7
Is Research Needed ?
No
9
Yes
Research Studies
Were Objectives Met ?
No
Yes
8

Figure 5. (Continued)

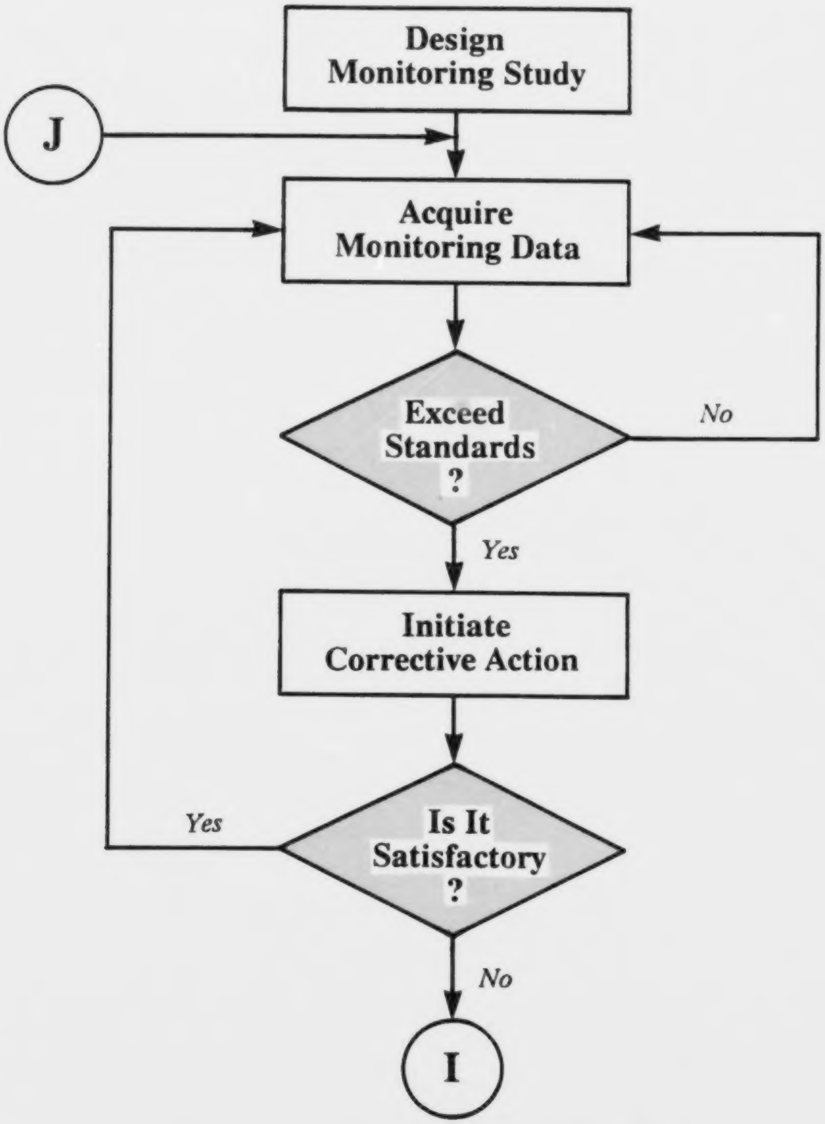
Design
Monitoring Study
J
Acquire
Monitoring Data
Exceed
Standards
?
No
Yes
Initiate
Corrective Action
Is It
Satisfactory
?
Yes
No
I

Figure 6. Monitoring data flow.

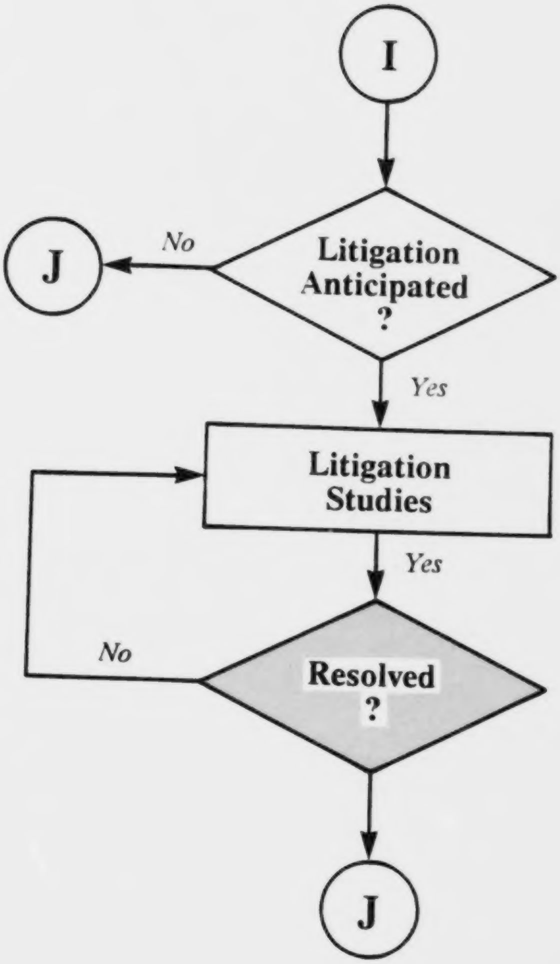
I
No
J
Litigation Anticipated ?
Yes
Litigation Studies
Yes
No
Resolved ?
J

Figure 6. (Continued)

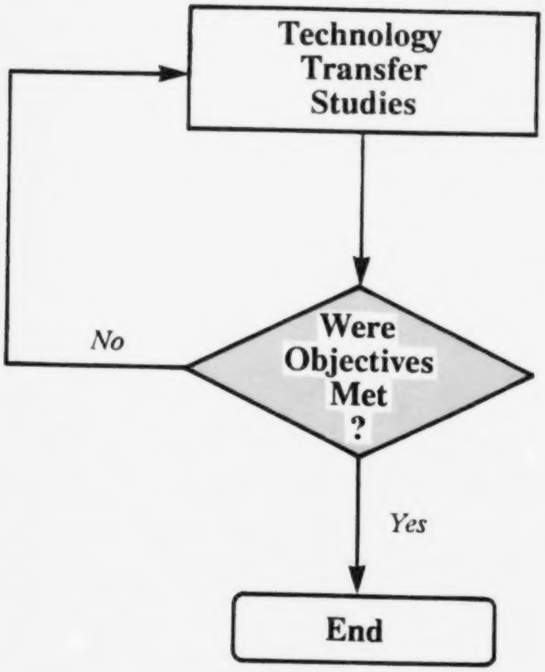
Technology Transfer Studies
Were Objectives Met ?
No
Yes
End

Figure 7. Technology transfer data flow.

The main area where the magnitude of the soil sampling can be controlled is in the precision required by the sampling designs. The accuracy of the sampling is unknown because the true average is not available. Repeated sampling for high precision nevertheless, must rely on the analytical accuracy obtained in the laboratory to insure that the methods used measure what is present in the soil sample. Thus, the accuracy for analysis applies only to the samples, while the accuracy for the study depends on the degree to which the samples are representative of the area. The balancing of resources with data reliability is a primary goal of the DQO process.

The specific goals for each type of study will determine the allowable probabilities of Type I and Type II errors and the minimum relative difference between sampled population mean and either background mean, or designated action level that is considered important to detect. Suggested guidelines are presented below for the operational situations listed previously.

PRELIMINARY SITE INVESTIGATION

The preliminary investigation is the foundation upon which other studies in hazardous waste site assessments should be based. As part of this study, it is essential to determine whether or not soils are the sample media of importance to the total assessment. The total assessment must provide data which will enable decision makers to decide whether the soil contaminants pose an imminent and substantial endangerment to human health requiring emergency action, and whether there is an unacceptable long-term risk to man or the environment. If soils are determined to be unimportant in the preliminary study, it is likely that no further attention will be directed to them. In view of this, a Type II error is considered to be of greater importance than a Type I error. Presented below are suggested guidelines for use in developing DQOs that may be used initially.

Confidence Level $(1-\alpha)$	Power $(1 - \beta)$	Relative Increase over Background $[100(\mu_s-\mu_B)/\mu_B]$ to be Detectable with a Probability $(1 - \beta)$
70 - 80%	90 - 95%	10 - 30%

If resources limit the number of samples that can be taken, the investigator should determine, for the number of samples that can be collected, value judgment-based optimum values for confidence level, power, and detectable relative difference. If these values are deemed adequate, the study may proceed.

Using five-percent duplicate samples may provide adequate QA/QC for measuring variance between samples (Plumb, 1981). However, there should be a minimum of two sets of duplicates in each strata sampled. As data become available, these assumptions should be checked. This is usually accomplished by collecting and analyzing more duplicates initially and then checking to determine the minimum number required for the sites being sampled and the pollutants being measured.

EMERGENCY CLEAN-UP

Emergency sampling is designed to identify those areas in which soils are contaminated to such a degree as to threaten imminent and substantial endangerment to human health. The threat may be due to the soils acting as a source of hazardous constituents to drinking water, air, or human foods. The emergency action in these cases may be nothing more than staying indoors on windy days, using dust suppression, switching to bottled water for drinking and/or taking certain locally produced human foods off the market rather than a full-scale soil removal program. Soil removal may well be implemented at a later date as part of a planned removal or a remedial response operation. Of course, any long-term solution to the problem would also have to address the removal of the primary source of hazardous substances to the soils.

For an emergency response operation involving soils, a Type II error is considered of greater importance than a Type I error. Presented below are suggested guidelines for developing DQOs to be used for emergency response operations.

Confidence Level $(1 - \alpha)$	Power $(1-\beta)$	Relative Increase over Background or an Action Level to be Detectable with Probability $(1-\beta)$
80 - 90%	90 - 95%	10 - 20%

PLANNED REMOVAL AND REMEDIAL RESPONSE STUDIES

Planned removal and remedial response studies are sometimes continuations of those initiated during emergency clean-up studies. They should be designed to provide specific information needed to resolve control option issues. The areas to be surveyed should be stratified and sampled according to a design that can be used to determine spatial variability. A suitable statistical design should be formulated so that components of variance for the study situation may be identified and evaluated. Appropriate QA/QC procedures must be formulated and implemented.

If the sampling during exploratory or emergency response investigations has been done properly, there will be a sound basis for determining the sample size and sampling site distributions. The design will have to incorporate information on the vertical distribution as well as the horizontal distributions. Measurements of concentration trends with time may be of critical importance, particularly if soil concentrations are changing appreciably with time. For example, the concentrations of pollutants in soils may decrease with time once the primary source of contamination is removed. This reduction in concentration may be due to a combination of biotic degradation of the contaminants, chemical degradation, volatilization, removal of contaminants by leaching, etc.

For a planned removal or a remedial response operation involving soils, it is considered that a Type I and a Type II error are of about equal significance. Furthermore, an attempt at cost recovery which might lead to litigation is a likely successor to these studies. Accordingly, it is important to achieve the highest order of precision and accuracy feasible. Presented below are suggested guidelines for developing DQOs that may be used for planned removal and remedial response studies.

Confidence Level $(1 - \alpha)$	Power $(1-\beta)$	Relative Increase over Background or an Action Level to be Detectable with Probability $(1-\beta)$
90 - 95%	90 - 95%	10 - 20%

MONITORING OR RESEARCH STUDIES

The guidelines for confidence levels, power, and detectable relative differences for monitoring or research studies should be set on the basis of the objectives of each study. As actions which may be taken on the basis of resulting data become more and more significant and costly, greater effort should be placed on achieving an increased level of reliability for the data. Publication of the results in a peer-reviewed journal will usually require some demonstration that an adequate QA/QC plan has been incorporated into the experimental protocol.

CHAPTER 8

SELECTION OF NUMBERS OF SAMPLES AND SAMPLING SITES FOR THE DEFINITIVE STUDY

INTRODUCTION

The QA/QC plan must be designed to allow for estimation of errors in the determination of, as a minimum, mean concentrations and standard deviations of the means. In some cases the primary interest may be in the determination of a reasonable mean of extreme values (the stratum having the highest mean concentration) which must be compared to an acceptable action level. In the latter case corrective actions will generally be required if the acceptable action level is deemed to be exceeded. For this case, the QA/QC plan must provide data on the basis of which one may state with what reliability the action level is, or is not, exceeded. Both Type I and Type II errors must be taken into consideration. These errors can be controlled only by choosing an appropriate number of samples. (See Table 4.)

On the basis of data from the exploratory study, the following minimum amount of information will be available.

TABLE 4. NUMBER OF SAMPLES REQUIRED IN A ONE-SIDED ONE-SAMPLE t-TEST TO ACHIEVE A MINIMUM DETECTABLE RELATIVE DIFFERENCE AT CONFIDENCE LEVEL (1-α) AND POWER OF (1-β).

Coefficient of Variation (%)	Power (%)	Confidence Level (%)	Minimum Detectable Relative Difference (%)				
			5	10	20	30	40
10	95	99	66	19	7	5	4
		95	45	13	5	3	3
		90	36	10	3	2	2
		80	26	7	2	2	1
	90	99	55	16	6	5	4
		95	36	10	4	3	2
		90	28	8	3	2	2
		80	19	5	2	1	1
	80	99	43	13	6	4	4
		95	27	8	3	3	2
		90	19	6	2	2	2
		80	12	4	2	1	1
15	95	99	145	39	12	7	5
		95	99	26	8	5	3
		90	78	21	6	3	3
		80	57	15	4	2	2
	90	99	120	32	11	6	5
		95	79	21	7	4	3
		90	60	16	5	3	2
		80	41	11	3	2	1
	80	99	94	26	9	6	5
		95	58	16	5	3	3
		90	42	11	4	2	2
		80	26	7	2	2	1
20	95	99	256	66	19	10	7
		95	175	45	13	9	5
		90	138	36	10	5	3
		80	100	26	7	4	2
	90	99	211	55	16	9	6
		95	139	36	10	6	4
		90	107	28	8	4	3
		80	73	19	5	3	2
	80	99	164	43	13	8	6
		95	101	27	8	5	3
		90	73	19	6	3	2
		80	46	12	4	2	2

TABLE 4. CONTINUED

Coefficient of Variation (%)	Power (%)	Confidence Level (%)	Minimum Detectable Relative Difference (%)				
			5	10	20	30	40
25	95	99	397	102	28	14	9
		95	272	69	19	9	6
		90	216	55	15	7	5
		80	155	40	11	5	3
	90	99	329	85	24	12	8
		95	272	70	19	9	6
		90	166	42	12	6	4
		80	114	29	8	4	3
	80	99	254	66	19	10	7
		95	156	41	12	6	4
		90	114	30	8	4	3
		80	72	19	5	3	2
30	95	99	571	145	39	19	12
		95	391	99	26	13	8
		90	310	78	21	10	6
		80	223	57	15	7	4
	90	99	472	120	32	16	11
		95	310	79	21	10	7
		90	238	61	16	8	5
		80	163	41	11	5	3
	80	99	364	84	26	13	9
		95	224	58	16	8	5
		90	164	42	11	6	4
		80	103	26	7	4	2
35	95	99	775	196	42	25	15
		95	532	134	35	17	10
		90	421	106	28	13	8
		80	304	77	20	9	6
	90	99	641	163	43	21	13
		95	421	107	28	14	8
		90	323	82	21	10	6
		80	222	56	15	7	4
	80	99	495	126	34	17	11
		95	305	78	21	10	7
		90	222	57	15	7	5
		80	140	36	10	5	3

- Mean concentrations and standard deviations of the means for stratified regions (assuming it was deemed necessary to stratify the study region).

- Mean concentrations and standard deviation of the mean for the control region.

- Results of tests at specified confidence levels to determine whether or not the mean concentrations in all strata are significantly different from the control region mean concentration.

- Results of tests at specified confidence levels to determine whether or not peak or maximum measured concentrations exceed any established action levels.

- Some measure, through analysis of variance tests, of the distribution of observed variances among various elements of the sampling process such as sample collection, sample handling, and sample analysis.

An evaluation must be made during Stage 2 of the DQO process to determine which elements of the exploratory study provide sufficient information to meet program objectives, and where additional measurements will be necessary. Generally, since the exploratory study was designed to provide only a limited sample of the desired study population, it will be necessary to obtain additional measurements to improve the levels of precision and confidence, to confirm the results, and to expand the measurements to cover regions not previously sampled.

NUMBER OF SAMPLING SITES REQUIRED

The minimum number of samples, n, required to achieve a specified precision and confidence level at a defined minimum detectable relative difference may be estimated by the use of Table 4 or one of the following equations:

$$n \geq [(Z_\alpha + Z_\beta)/D]^2 + 0.5Z^2_\alpha$$

for a one-sided, one-sample t-test, and

$$n \geq 2[(Z_\alpha + Z_\beta)/D]^2 + 0.25Z^2{}_\alpha$$

for a one-sided, two-sample t-test

where: Z_α is a percentile of the standard normal distribution such that $P(Z \geq Z_\alpha) = {}_\alpha$, Z_β is similarly defined, and D = (minimum relative detectable difference/CV. CV = coefficient of variation. For a two-sided t-test, the values for Z_α should be changed to $Z_{\alpha/2}$.

As an example of application of the first equation above, assume CV = 30%, Confidence Level = 80%, Power = 95%, and Minimum Detectable Relative Difference = 20%. From Appendix B for infinite degrees of freedom (t distribution becomes a normal one) $Z_\alpha = 0.842$ and $Z_\beta = 1.645$. From the data assumed, D = 20%/30%. Therefore

$$n \geq [(0.842 + 1.645)/(20/30)]^2 = 0.5\ (0.842)^2$$

$$n \geq 13.917 + 0.354 = 14.269$$

n = 15 (always round up) which agrees with the value given in Table 4.

In case multiple pollutants are present, the particular pollutant requiring the greatest number of samples to achieve the assigned DQOs would be the controlling factor. In this instance, however, all samples collected may not have to be analyzed for all pollutants.

In general, a suitable soil sample from a number of possible sampling designs may be selected on the basis of random, stratified random, judgmental, or systematic sampling. The authors recommend the use of geostatistical techniques as the most appropriate methods of handling spatial data. The tools of geostatistics are easier to apply and the utilization of resources is better if one of the systematic designs is used.

The optimum approach appears to be a combination of systematic and judgmental sampling. Assuming that appropriate information has been obtained in the information-gathering phase of the exploratory study, a conceptual model may be hypothesized describing the spatial distribution of soil contamination, as well as identifying a likely background or control area. Judgmental samples can be taken for any purpose; however, these purposes must be documented and explained. Randomization of a systematic grid can be difficult because, once the spacing is selected, the starting point identified and the orientation chosen, there are no degrees of freedom to use for randomizing the sample location.

The major axis of orientation should be selected along the most likely route of migration. It is likely that there will be no indication of the most likely direction of migration; however, in such cases, the direction used as the major axis of the sampling grid can be chosen at random.

The starting point of the grid may be chosen on the basis of either a random start or the center of the pollution source. A smelter stack would be a possible point source for the pollution, and could be the starting point for the grid. The major axis would be the direction of the prevailing winds. Sites with no known major concentration(s) or where the source is quite large in area require a different starting point. This can be handled by placing a grid over a map of the area. A random number table can then be used to select a grid point on this map, which becomes the starting point for the sampling grid. Once the sampling grid is properly transferred to the map, a convenient numbering scheme can be set up to allow identification of the samples.

Samples would then be collected from each of the grid nodes or at some subset of these nodes, depending upon the intensity of the sampling expected. The Palmerton NPL Site study, discussed previously, provides a good example of this approach. Samples were collected intensely in the areas near the smelter sites, but only selected nodes were sampled in areas distant from the sources. The starting point for the grid was the center of the Town of Palmerton.

CHAPTER 9

CONTROL OF MEASUREMENT-ERROR VARIANCE

INTRODUCTION

The quality assurance plan should address two types of variation in soil sample data. One is the population variation, the variation between true sample values, which is a function of the spatial variation in the pollutant concentrations. Treatment of this type of variation is discussed in Chapter 10. The other variation, measurement-error variation in the data, is induced by differences between true sample values and reported values.

The distribution of the true values of the pollutant concentrations in the population of sampling units will typically be multimodal and nothing like the probability distributions dealt with in statistics textbooks. The modes of the distribution will probably correspond to background values, and concentrations of various types of materials that have found their way to the site being sampled. It is the distribution of measurement errors and of deviations of true values from expected values (see Geostatistics, Chapter 10) that are of principal concern in the development of the quality assurance plan and in the evaluation of the quality assurance data. Fortunately, these latter distributions generally are similar to distributions discussed in statistics texts.

Plans for the taking of samples, analysis of samples, and analysis of resulting data are based on assumptions concerning the probability distributions of the measurement errors or of the deviations of true values from expected values. These assumptions should be consistent with results from past surveys taken under similar conditions, and, in particular, with the results of an exploratory study.

The variability in measurement errors is a function of the variable being measured, the sample collection method and handling procedures, the analytical procedure, and the data transcription procedures. If the distribution of the measurement errors is normal, it is symmmetric about its expected value (center of gravity of the probability distribution), and its variability is completely characterized by its variance (moment of inertia of the probability distribution about its center of gravity when probability is treated as mass). The symmetry makes the expected value a reasonable measure of location, whereas in non-symmetric distributions other measures of location may be preferred (e.g., the median). Also, the statistician has means of dividing variance into components representing various sources of variation.

With most non-normal probability distributions, the variability is only partially described by the variance. Hence, these properties of symmetry, and variance representing variation, are two prime reasons for transforming variables so that the new variables will have approximately normal probability distributions. Procedures for such transformations are given in Box and Cox (1964) and in Hoaglin *et al.* (1983). A discussion of the importance of the normality assumption and data transformations appears in Scheffe (1959). In what follows, we shall assume that the data have been transformed so that the measurement errors are nearly normal in distribution. Additional information about the distribution of measurement errors for various types of pollutants and measurement procedures may be obtained from the U.S. EPA's Regional Offices and Laboratories and its National Enforcement Investigation Center in Denver, Colorado.

If the variable of interest has a count measurement, such as with radioactivity or the presence or absence of a pollutant, other statistical methods are required. These methods are usually denoted as qualitative or discrete statistical methods. Bishop *et al.* (1975) is a good reference to such procedures. The methods of this chapter should not be applied to count data.

As stated above, the measurement error variance is the variance of the differences between the true concentrations for the sampling units and the reported concentrations. The variance of the differences between the true and reported concentrations is typically the sum of variances of many random errors that are made in sample taking, sample handling, sample analysis, and data transcription.

GOALS

There are two commonly encountered rules of thumb in restricting the measurement-error variance that may be viewed as goals in quality assurance. They are quite similar and equally reasonable. One rule says keep the measurement error variance to less than one-tenth the total variance between measurements; the other says keep the measurement error standard deviation to less than one-fourth the total between-measurement standard deviation.

The reason for these rules is that if they are achieved in the absence of measurement bias, the measurement error on the average is so small relative to the differences between measurements that it can effectively be ignored when analyzing the data. If one has not accomplished this goal in a survey, there are many almost insoluble difficulties in the final analysis of the data. A major problem is that measurement errors are typically correlated (e.g., a calibration error that causes one measurement to be too large will cause the other measurements to be too large until the next calibration). The levels of correlation in these measurements are difficult to estimate, but are needed to calculate estimation variances if measurement error variance is not small relative to total variance. Once either goal has been reached, it is hard to justify any additional effort to reduce measurement error variance, since that reduction will affect such a negligibly small change in the total variance and in the variances of the sample estimates.

It is necessary to search out the major sources of measurement error variance and develop QA procedures to ensure that these sources are controlled. It is also necessary that QA data be obtained to monitor the sources of error and to provide an estimate of their contributions to total error variance in the final QA data analysis.

COMPONENTS OF VARIANCE

The measurement error in a soil sampling survey is usually the sum of several errors from independent sources. The total measurement error variance can be represented by a sum of the variances of the errors arising from these independent sources. A procedure called components of variance analysis (Scheffe, 1959; Snedecor and Cochran, 1982) provides estimates of the portion of the total variance coming from each of the sources in the measurement process. Basic assumptions of this procedure are that the measurement errors are normal in distribution, independent, and for each independent source have constant variance.

> Example: Consider the hypothetical data from a stratified random sample design that has four strata, three random samples per stratum, two subsamples per sample, and one analysis per subsample. The stratum effects are assumed to be fixed unknown constants. The random sources of variation in the data are between samples within strata and between subsamples within samples (combined with analytical error). In the table of data below, a period in place of a letter in the subscript means that the data have been summed over that letter (e.g., $\Sigma_k X_{ijk} = X_{ij.}$).

Computations:

I. $C = X_{...}^2/(abn) = (82.30)^2/24 = 282.2204$

II. Total: $\Sigma_i \Sigma_j \Sigma_k X_{ijk}^2 - C = (3.17^2 + ... + 4.63^2) - C = 29.8656$

III. Strata: $\Sigma_i X_{i..}^2/bn - C = (14.75^2 + ... + 26.81^2)/6 - C = 23.7517$

IV. Samples: $\Sigma_i \Sigma_j X_{ij.}^2/n - C = (5.81^2 + ... + 20.42^2)/2 - C = 26.3109$

V. Samples within Strata: IV - III = 26.3109 - 23.7517 = 2.5592

VI. Subsamples within Samples: II-IV = 29.8656 - 26.3109 = 3.5547

These computations are now organized within the table below.

Analysis of Variance

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	Expected Mean Square
Strata	a-1=3	23.7517	7.9172	$\sigma_A^2 + n\sigma_B^2 + bnM/3$
Samples/ Strata	a(b-1)=8	2.5592	0.3199	$\sigma_A^2 + n\sigma_B^2$
Subsamples/ Samples	ab(n-1)=12	3.5547	0.2962	σ_A^2
Total	23	29.8656		

From the analysis of variance table, one obtains the variance estimates:

$s_A^2 = 0.2962$, which estimates σ_A^2, variance owing to subsampling and analysis;

$s_B^2 = (0.3199 - 0.2962)/2 = 0.0118$, which estimates σ_B^2, the variance owing to sampling within strata.

The symbol M in the above table stands for the sum of squared deviations of stratum means about their grand mean.

The results of this analysis indicate that the experimenter should either have made a greater effort to reduce subsampling and analytical errors or taken many more subsamples, since the error variance estimated by s_A^2 (= 0.2962) is much larger than the estimated variance s_B^2 (= 0.0118), between samples within strata, which is just the opposite of the goal suggested earlier in this chapter.

While the above example illustrates a classic components of variance analysis for a situation in which the data have a hierarchical structure (i.e., strata, samples within strata, etc.), there are many instances in environmental monitoring where this hierarchical structure is lacking and other methods of separating the variance components are called for. Such an example involving quality assurance samples is given in the next section.

QA SAMPLES

In quality assurance, procedures are specified for the survey in an attempt to keep measurement errors, measurement bias, and measurement error variance small. It is essential that the sampling project include the means to determine whether the procedures are being followed and the necessary data at the end of the survey to show how successful the quality assurance procedures were in controlling measurement-error variance. To obtain the needed data, it is necessary to introduce QA/QC samples into the measurement process.

The principal independent sources of random error must be specified. If the independent sources of random error are listed as sources A, B, ..., then the total measurement error variance, σ_T^2 can be written as

$$\sigma_T^2 = \sigma_A^2 + \sigma_B^2 + \ldots,$$

where σ_A^2 is the variance of the random errors associated with source A, etc. A partial list of such sources might include failing to sample at specified sampling locations, mistakes in taking the sample, errors in processing the sample, subsampling errors, analytical errors, and transcription errors. Measurements on quality assurance samples can be used to estimate the variances from one source or the combined variance of several sources of random measurement error. In addition, quality control samples can be used to determine whether the measurement systems are in control during the survey. For example, laboratory audit samples are run along with field samples. If the errors in the measurements of these audit samples are too large, they will indicate that the laboratory analytical process is out of control and that corrective action is required prior to analysis of additional field samples.

> Example: This example again considers the exploratory study performed at the Palmerton NPL Site (Starks *et al.*, 1987) mentioned earlier. The duplicate samples (Table 2) taken at each of ten sampling locations were QC samples. The individual cores (Table 3) that were taken at ten sites but not composited were QC samples. In this study, the four composited cores taken from each site were mixed and sieved. A subsample was then taken and sent to the laboratory for analysis of concentrations of four metals (Cd, Pb, Cu, and Zn). For the soil from 10 sites, an additional subsample (called a split) was taken after the mixing and sent to the laboratory with no identification to associate it with the first subsample taken from the soil sample. These splits were also QC samples. The results from the splits are given in Table 5.

This example shows how these three types of QC samples were used in evaluating the QA procedure employed in the exploratory study.

The variance estimate $s^2 = 0.0032$ in Table 5 gives an estimate of the measurement error variance coming from subsampling, analysis, and data recording. The variance estimate $s^2 = 0.0691$ obtained from duplicate samples in Table 2 is an estimate of the total of the variance coming from short range (0.5 m) spatial variation, sample taking and handling, sifting and mixing, and also the subsampling, analysis, and data recording. Hence, the difference, $s_d^2 = 0.0659$, between these two variance estimates is an estimate of the total of the measurement error variances coming from short range (0.5 m) spatial variation, sample taking and handling, sifting and mixing. If the variance s_d^2 were primarily from errors in sampling handling, sifting and mixing, one would expect a variance between individual cores ($s^2 = 0.3659$, Table 3) similar to that between duplicates. This was not the case, so one is led to the conclusion that the combination of short-range spatial variation and variation in sample taking is the major contributor to total measurement-error variance. For this reason, the support of the sampling units was increased from four to nine cores in the second (definitive) study.

TABLE 5. RESULTS FROM SPLITS AT THE PALMERTON NPL SITE

SITE	CADMIUM[a]		D[b]	L[b]
DD70	4.17	4.13	0.04	0.010
BV35	116	106	10.00	.090
CB34	73.8	63.1	10.70	.157
BT70	7.34	7.32	0.02	.003
AY34	88.0	81.9	6.10	.072
BT35	95.1	83.1	12.00	.135
DP82	2.50	2.43	0.07	.028
CB42	280	279	1.00	.004
BR34	68.7	63.3	5.40	.082
CD24	9.1	8.99	0.11	.012

$$s^2 = \Sigma L_i^2/20 = 0.0032$$

[a] units are mg/kg

[b] D = absolute pair difference, and L = absolute pair difference of log-transformed data

Table 6 lists typical QA/QC samples and how measurements of these samples are used in the control of the measurement process and in the evaluation of the quality assurance procedures employed by the project. To obtain an unbiased measure of the internal consistency of the samples and their analyses, the individual QA/QC samples should be labeled with a code number so that the chemist (and preferably also the laboratory) does not know the relationship between the samples he is analyzing. This reduces the chances of conscious or unconscious efforts to improve the apparent consistency of the analyses.

Samples can be split to:

- provide samples for both parties in a litigation or potential litigation situation;
- provide a measure of the within-sample variability;
- provide materials for spiking in order to test recovery; and
- provide a measure of the analytical and extraction errors.

The location of the sample splitting determines the components of variance that are measured by the split. A split made in the sample bank (i.e., facility to which samples are sent from the field) measures error introduced from that level onward. A split made in the field includes errors associated with field handling. A split or series of subsamples made in the laboratory for extraction purposes measures the extraction error and subsequent analytical errors.

Table 6. Type of QA/QC Samples or Procedures

Procedure	Description
1. Field Blank	A sample container filled with distilled, deionized (DDI) water, exposed during sampling and then analyzed to detect accidental or incidental contamination.
2. Sample Bank Rinsate	A sample (last rinse of DDI water) of DDI water, passed over the sample preparation apparatus, after cleaning, to check for residual contamination.
3. Field Rinsate	A sample (last rinse of DDI water) of DDI water, passed over the sampling apparatus after cleaning, to check for residual contamination.
4. Reagent Blank	A DDI water sample analyzed as a routine sample to check for reagent contamination.
5. Calibration Check Standard	A standard material to check instrument calibration.
6. Spiked Extract	A separate aliquot of extract to which a known amount of analyte is added to check for extract matrix effects on the recovery of added analyte.
7. Spiked Sample	A separate aliquot of the soil sample having an appropriate standard reference material added to check for soil and extract matrix effects on recovery.
8. Total Recoverable	A second aliquot of the sample which is analyzed by a more rigorous method to check the efficacy of the protocol method.
9. Laboratory Control Standard	A sample of a soil standard carried through the analytical procedure to determine overall method bias.
10. Re-extraction	A re-extraction of the residue from the first extraction to determine extraction efficiency.

(Continued)

Table 6. Continued

Procedure	Description
11. Split Extract	An additional aliquot of the extract which is analyzed to check injection and instrument reproducibility.
12. Triplicate Samples (Splits)	The prepared sample is split into three portions to provide blind duplicates for the analytical laboratory and a third replicate for the referee laboratory to determine interlaboratory precision.
13. Duplicate Sample	An additional sample taken near the field sample to determine total within-batch measurement error.
14. Field Audit	A sample of well-characterized soil that is taken into the field with the sampling crew, sent through the sample bank to the laboratory with the field samples to detect bias in the entire measurement process and to determine batch to batch variability.
15. External Laboratory Audit	A sample of well-characterized soil sent directly to the laboratory for analysis. The analyte concentrations are unknown to the laboratory. This type of sample is used to estimate laboratory bias and batch-to-batch variability. It may also be used for external quality control of the laboratory.
16. Internal Laboratory Audit	A sample of well-characterized soil, whose analyte concentrations are known to the laboratory, to be used for internal laboratory quality control.

Spiked samples are prepared by adding a known amount of reference chemical to one of a pair of split samples. Comparing the results of the analysis of a spiked member to that of the non-spiked member of the split measures spike recovery and provides a measure of the analytical bias. Spiked samples are difficult to prepare with soil material itself. Frequently the spike solution is added to the extract of the soil sample. This avoids the problem of mixing, but does not provide a measure of the interaction of the chemicals in the soil with the spike, neither does it provide an evaluation of the extraction efficiency.

Blanks and rinsates provide a measure of various cross-contamination sources, background levels in the reagents, decontamination efficiency, and other potential error that can be introduced from sources other than the sample. For example, a field blank measures input from contaminated dust or air into the sample. A rinsate sample measures any chemical that may have been on the sampling tools after the decontamination process is completed.

A question that frequently arises is how many QA/QC samples of each type are needed in a study. One often sees rules of thumb such as one for every 20 field samples. However, such rules of thumb are oversimplifications and should be treated with great caution. A better approach is to determine how each type of QA/QC sample is to be employed and then determine the number for that type based on the use. For example, field duplicates are used to estimate the combined variance contribution of several sources of variation. Hence, the number of field duplicates to be obtained in the study should be dictated by how precise one wants that estimate of the variance to be. The precision of an estimate of the variance depends on the degrees of freedom (i.e., number of duplicate pairs) of the estimate. Table 7 gives the 95% confidence intervals for various numbers of degrees of freedom, based on an assumption that the data are, or have been transformed to, normally distributed data. Methods for obtaining such confidence intervals for any number of degrees of freedom are given in most elementary statistics texts.

Table 7. Some 95 Percent Confidence Intervals for Variance

Degrees of Freedom	Confidence Interval
2	$0.27s^2 \leq \sigma^2 \leq 39.21s^2$
5	$0.39s^2 \leq \sigma^2 \leq 6.02s^2$
10	$0.49s^2 \leq \sigma^2 \leq 3.08s^2$
20	$0.58s^2 \leq \sigma^2 \leq 2.08s^2$
50	$0.70s^2 \leq \sigma^2 \leq 1.61s^2$
100	$0.77s^2 \leq \sigma^2 \leq 1.35s^2$

If it is decided that 20 degrees of freedom gives satisfactory precision for the estimate of the variance, one might equally space the duplicate samples among the field samples so as to have 20 duplicates by the end of the survey. Alternatively, one might take duplicate samples at a fairly high frequency at the start of the survey until 10 duplicate pairs are obtained and then obtain the remaining ten duplicate pairs at a reduced rate over the remainder of the survey. This second procedure would allow an early estimate of the variance based on 10 degrees of freedom to determine whether the QA plan is resulting in error variances in the range expected, and the remaining ten pairs would allow the after-survey variance estimate to take the entire survey into account.

Some types of samples, such as the calibration check standards, are used to provide a quality control function. That is, if measurements of these check standards differ by too much from their reference values, the instrument is declared out of control and will have to be adjusted. Then it will be necessary to go back and re-analyze all samples between the last in-control reading and the out-of-control reading. The frequency of use of samples of this quality control type should be based on costs of the analyses of these samples versus the costs of reanalyzing field samples in out-of-control situations. This frequency of use will also be a function of the probability of obtaining an out-of-control situation in the laboratory. Of course the objective is to minimize expenditures of both time and money while obtaining data of adequate quality.

The percentage of the total monitoring effort allocated to QA/QC will depend on many factors including the size of the project, the available knowledge concerning sampling and analytical procedures, the relationship of environmental risk to pollutant concentration, and the nearness of action levels to method detection limits. Typically the smaller the project, the larger will be the proportion of cost allocated to QA/QC. New, untried procedures will typically require pilot study runs and additional training for personnel. If the action level is near the method detection limit, there will be little room for error in the measurements, and the QA/QC effort may have to be large to assure that measurement errors are kept small. One should not specify a certain percentage of a project's costs to QA/QC without considering the above factors.

BIAS

Bias identifies a systematic component of error that causes the mean value of the sample data to be either consistently higher or consistently lower than the true mean value. Bias may be caused by faults in sampling design, sampling procedure, handling procedure, or analytical procedure. An example of a bias would be the error in analytical results introduced by an instrument's being out of calibration during a portion of the analysis. Laboratories usually introduce reference and audit samples into their sample load to detect possible changes. Bias in soil sampling is difficult to detect. The presence of a bias can be proven by the technique described as standard additions or by using audit samples. On the other hand, it is difficult to prove that bias is not present because an apparent lack of bias may be the result of an inability to measure it rather than its actual absence.

A procedure called standard additions is commonly used to detect bias in a sampling effort. In this procedure, known amounts of standard solutions are added to aliquots of soil samples. It is recommended that this be done in the field or in a field laboratory. The main problem encountered is that mixing soils to obtain homogeneity is difficult in a laboratory, and even more so in the field. Several known quantities of the standard are added to the aliquots of the soil samples. The analytical results should follow a straight line:

$$y = a + bx,$$

where x is the increase in concentration caused by the addition and y is the value obtained by the laboratory. Bias is indicated if the data do not follow a straight line, or if $a < 0$. If the units of x and y are the same, the value of b should be near one, and a significant deviation from one would indicate a proportional bias.

CHAPTER 10

SAMPLE DESIGN AND DATA ANALYSIS

Data obtained from soil sampling is used to estimate characteristics of the sampled population, such as pollutant concentrations on action supports at various locations on the study site and mean concentrations of background regions. There are three basic approaches for increasing the precision of statistical estimates and the power of statistical tests to be based on survey results. They are: (1) to use more efficient statistical estimators and tests; (2) to improve the sampling design; and (3) to increase the sample size (i.e., to increase the sampling density). This chapter deals with the influence of sample design and estimation techniques on the variance of estimates and the power of tests, with determination of required sampling density, and with statistical analysis of survey data.

SAMPLE DESIGN

Given a site to be sampled, several decisions must be made as to how the soil will be sampled. First, the support for the sampling unit must be specified, then decisions concerning type of sampling design and sampling density must be made. The prime objectives of a statistical sampling design are either to provide the most complete information possible about a question of interest for a fixed survey cost or to minimize survey cost for a fixed amount of information. A common measure of the amount of information provided by a survey about an estimated parameter is the inverse of the variance of the estimate. Secondary concerns in sampling design are simplicity of resulting data analysis and simplicity of field operations in performing the survey.

A common type of design given in many elementary texts is the simple random sample design in which the sampling units are determined by random selection without replacement. This plan for site investigation simplifies the statistical analysis; however, it is typically very wasteful of resources and is, therefore, very difficult to justify.

The stratified random design is another common type of design. With this design the region to be sampled is partitioned into subregions (strata) on the basis of suspected differences in level of pollutant, on cost of sampling, on the basis of equal strata areas, or on some combination of the above. A simple random sample is taken from each stratum. For example, one may have sufficient information to divide the site into strata where the level of pollutant concentrations is either far above action level, near action level, or far below action level. In this case, it would seem reasonable to expend most of the sampling effort (i.e., high sample density) on the strata that is near action level, so that one can decide with a high level of accuracy which parcels of land need remediation and which do not. Stratification ensures that all subregions of the site will be sampled, which may not be the case with a simple random sample of the site.

Stratification can use scientific or historical knowledge that the pollutant concentrations are quite different in identifiable segments of the area being sampled to improve the subsequent estimate of the mean concentration over the entire site. Another criterion that may be useful in stratification for environmental soil sampling is distance from known point sources.

Both stratified random sample and simple random sample procedures were developed for sampling of discrete sampling units and do not adequately take into account the spatial continuity and spatial correlation of soil properties. Samples taken at locations that are close together tend to give redundant information and are therefore wasteful of resources. For this reason, some type of sample selection grid (systematic design) is often used to assure that sample locations will not be close to one another. The grid may be radial, triangular, rectangular, hexagonal, etc.

Systematic grid designs provide many of the advantages of stratification plus the avoidance of redundant samples. They thereby improve precision and power. Investigations of the efficiencies of the grid designs show that the hexagonal grid is the most efficient given certain assumptions about the spatial distribution of the pollutant, but the square or rectangular grid is easier to use in practice. The difference in efficiency is not great. The radial grid has some advantages in investigating the distribution of a pollutant near a point source.

A grid will typically be oriented in the direction of flow of the pollutant, which may relate to site topography or a wind rose. Once the sampling density (grid spacing) and the orientation of the grid has been determined, a selection of one sample location will completely determine the locations of all sample locations.

A possible shortcoming of the grid design is the possibility of a periodicity in the pollutant concentrations, with the grid spacing a multiple of the period. This is an extremely unlikely situation in pollution studies, but one way to guard against this possibility is to superimpose a small stratified random sample over the grid design. In this case the strata would be subregions of approximately equal area. In practice, even when a grid (systematic) design is employed, many of the actual sample locations will not be at the grid locations because of the presence of obstructions such as roads, houses, rocks, and trees. Also, a soil sample should not be taken at a specified grid point if it is evident that fill has recently been added or if there has been a recent grade cut at the location. When the field crew cannot sample at a specified location, they should have instructions to take a sample at the nearest point in a prespecified direction from the original point where a sample can be obtained, provided that the location is within a specified distance (usually less than half the grid spacing) of the original point.

Simple random sampling and stratified random sampling designs are among a class of designs originally developed for the sampling of units that are discrete objects such as people, houses, and retail stores. The statistical analysis techniques associated with these designs are primarily associated with the estimation of population means. The basic designs and statistical procedures associated with surveys of discrete objects are given in a text by Hansen *et al.*, (1953).

The systematic grid designs are more closely related to the sampling of continuous media such as soil, air, and sediment. In the sampling of continuous media, the sampling units must be defined in terms of support (Chapter 5). Gy (1982) gives an extensive description of techniques for the sampling of the continuous media of particulate materials. The statistical analyses associated with the results of these surveys of continuous media are typically aimed at estimating the spatial distribution of a property of the media such as a pollutant concentration or in finding "hot spots" within the region or site being sampled.

Many of the statistical techniques used in the analysis of data from surveys of continuous media fall into a category called geostatistics. For sample surveys involving random selection of sampling units, the statistical procedures are usually formed on a probability base provided by the randomization, while in geostatistics, the statistical inferences are based on what is known as a random field model. A good discussion of the nature and differences of these two approaches is given in a paper by Borgman and Quimby (1988).

ROLE OF QUALITY ASSURANCE IN SAMPLE DESIGN

The Quality Assurance Officer should be involved in reviewing the sampling design proposed by the investigator. He or she should require that the information obtained provides measures of the components of variance that are identified in the field. An additional quality check that should be undertaken as part of the QA program is the review of the design by qualified soil scientists and other peers who are in a position to provide the necessary oversight of the sampling effort.

Broms (1980) makes the following statement: "There should be a balance between the soil investigation method, the quality of the soil samples, and the care and skill spent on the preparation and testing of the samples. There is no point in spending time and money on careful sample preparation and testing if the quality of the samples is poor." The QA program must address the total flow of information from the design to the reporting of results. The sampling design is the foundation of the whole study; therefore, it must be given careful consideration if the purposes and data quality objectives of the sampling effort are to be met.

Compositing of Samples

From the point of view of geostatistics, it is desirable to have a sample support that has a fixed depth and a square horizontal cross-section, because such supports can be grouped together to form rectangular blocks and action supports, and potential clean-up areas (e.g., city lots) are typically rectangular. However, to sample such a square support of sufficient cross-sectional area to make its short-range variance small requires the taking of a very large quantity of soil. It is difficult to handle large quantities of soil and make them homogeneous prior to subsampling. One way to avoid this problem is to take a uniform array of soil cores within the square in sufficient number so that the error variance associated with the true differences between the average pollutant concentration of the squares and that of the associated composites of the cores is quite small relative to the short-range variance of pollutant concentrations of the square supports. This procedure is explained in detail in Starks (1986).

While compositing of cores at individual sampling sites can be quite advantageous in terms of handling costs and measurement errors, the compositing of samples from different sampling locations should be done with great caution if at all. The compositing of samples technique is often performed to reduce sample handling and analytical costs. This procedure is used extensively by agricultural workers to determine fertilizer requirements for farm fields. It is also done in medical studies to screen blood samples for relatively rare antibodies. Peterson and Calvin (1965) make the following statement about the technique:

> "It should be pointed out that the composite samples provide only an estimate of the mean of the population from which the samples forming the composite are drawn. No estimate of the variance of the mean, and hence, the precision with which the mean is estimated can be obtained from a composite of the samples. It is not sufficient to analyze two or more subsamples from the same composite to obtain an estimate of the variation within the population. Such a procedure would permit the estimation of the variation among subsamples within the composite, but not the variation among samples in the field. Similarly, if composites are formed from samples within different parts of a population, the variability among the parts, but not the variability within the parts, can be estimated. If an estimate of the variability among sampled units within the population is required, two or more samples taken at random within the population must be analyzed separately."

Youden and Steiner (1975) caution against the use of the composite sample for many of the same reasons as those outlined above. Since the prime purpose of QA/QC is to assess and assure acceptable values for the bias and the precision of the data and of estimates obtained from the data, it is essential to be able to gauge the precision of the data. Therefore, the compositing of samples cannot, in general, be recommended unless it is for a stated specific purpose and unless a justification is provided.

Some work on determining the precision of estimates of the mean from composite samples has been published. Such estimates of precision usually require strong assumptions about variance components and/or the stochastic nature of the composited samples. (See Duncan, 1962, and Elder *et al.*, 1980.)

GEOSTATISTICS

Geostatistics is an application of classical statistical theory to geological measurements that takes into account the spatial continuities of geological variables in estimating the distribution of variables. In many ways, geostatistics is for measurements taken in 2-, 3-, and 4-dimensional space (the three spatial dimensions and the time dimension), what time series is for measurements taken in one-dimensional space time. However, a principal use of time series is in forecasting; in geostatistics, the principal emphasis is on interpolation. Nevertheless, both statistical procedures emphasize modeling the process to get an insight into the system being investigated.

For purposes of discussion, consider sampling units that have a support that is a volume with a square horizontal cross-section (s x s) and a fixed depth d for a total volume of ds^2. It will be assumed that the correlation (and covariance) between a measurement of a sampling unit and that of any other sampling unit is strictly a function of the distance between the units. (It is important to remember that the population of all the sampling units is the volume of soil of interest in the site under investigation and that the soil samples taken in the survey form a proper subset of the population of sampling units.)

A geostatistical estimation procedure called **block kriging** (named after a South African mining engineer named D.G. Krige) is employed to estimate the mean pollutant concentration in a rectangular block of sampling units. The estimate of the mean concentration is a linear combination $\Sigma a_i z_i$ of the concentration measurements z_i obtained at sample locations on or near the block. The coefficients a_i are chosen to minimize, subject to certain constraints, the estimation variance $V(M^* - M)$ where M^* is the estimator of the mean block concentration and M is the mean block concentration.

$$V(M^* - M) = \Sigma\Sigma a_i a_j \mathrm{Cov}(Z_i,Z_j) + \Sigma\Sigma \mathrm{Cov}(C_h,C_k) - \Sigma a_i \Sigma \mathrm{Cov}(Z_i,C_h)$$

where Cov stands for covariance, Z_i is the random variable corresponding to measurement z_i, and C_h is the concentration of pollutant in sampling unit h in the block. The calculation of the coefficients a_i that minimize the estimation variance is a simple mathematical procedure involving the solution of a system of linear equations subject to a set of linear constraints on the a_i. Once the a_i are calculated, the estimate of the mean concentration and the variance for that estimator are found directly by substitution in the above equations. The constraints imposed on the coefficients a_i depend on how the expected value of Z_i is related to the location of the sampling unit i. The relationship between location and expected value is called **drift** in the geostatistical literature. If the expected value of Z_i is independent of the location of the sampling unit, one says that there is **no** drift. The nature of the drift and of the covariance function taken together are sometimes referred to as the **spatial structure** of the phenomenon being measured. Typically, the spatial structure will have to be estimated from the data, but the nature of the phenomenon being measured will usually provide basic information as to which spatial structures are reasonable and which are not. For example, in measuring the pollution emanating from a point source, it would seem reasonable to expect drift to be present; that is, one would expect to find higher concentrations of the pollutant close to the source than at a greater distance.

One of the major problems in using these geostatistical procedures is in the estimation of the covariance as a function of distance. It must be estimated from the data. The several ways of doing this, unfortunately, will typically give quite different answers. It is essential that the assumptions about drift used in obtaining an estimate of the covariance function be consistent with the realities of the phenomenon being measured and that the estimated covariance function be checked against the data for goodness of fit. This check against the data is done by a process called cross-validation. Cross-validation in this instance consists of kriging to obtain an estimate of the concentration at each sample location based on data from neighboring sample locations. The observed measurement at that location is then subtracted, and this difference is divided by the square-root of the estimation variance to obtain a standardized score. This is done for all sample locations, and the sample variance of the standardized scores is obtained. This sample variance should be close to one. (Starks and Fang [1982] also conjectured that the standardized scores should have an approximately normal distribution.) One of the problems faced by users of geostatistics is that there are a large number of software packages on the market that will take the data and do the kriging without any consideration of whether the assumptions implicit in the package procedure are correct and without any cross-validation of the results.

It should be pointed out that because kriging obtains estimates by assigning larger weights to nearby sample location measurements and smaller weights to those more distant, the estimations of pollutant concentrations are quite similar over a wide range of covariance functions that might be employed. However, in quality assurance, one is also interested in estimating the precision of the concentration estimates, and here is where the trouble lies. Bad estimates of covariance functions will usually lead to bad estimates of the precision of the kriging estimates. Bad estimates of precision in the exploratory study can, in turn, lead to inaccurate estimates of the number of samples needed in the definitive study.

Once an acceptable estimate of the covariance function has been found from the exploratory study, an acceptable spacing for sampling locations on a square grid can be determined by use of the kriging procedure. The estimation variance in block kriging is determined solely by the covariance function, the spacing of the sample locations, and the position of the block relative to the sample points. A block in the center of the square formed by four points in the sample location grid will have a larger estimation variance associated with it than will any other block of equal size located within the array of sampling locations. Hence, one can pick a grid spacing distance, arbitrarily assign values to the sample locations, perform the block kriging on a block at the center of a square, and find the maximum estimation variance for that block size. By trial and error, one can quickly find a grid spacing that gives a maximum estimation variance that is sufficiently small to be in accord with precision requirements of the quality assurance plan (i.e., satisfies data quality objectives).

WARNING: Kriging is a good procedure for interpolation, but a bad procedure for extrapolation. Do not give credence to block kriging estimates for locations that are beyond the range of the sample locations.

OBJECTIVES

In all the operational situations listed in Chapter 7, preliminary site investigations, emergency cleanup operations, planned removal operations, remedial response operations, monitoring, and research or technology transfer studies, one or all of the following questions will be of primary interest.

- Are there any action supports (see Chapter 5) within the study area that have pollutant concentrations above action level concentration?
- Where are the above-action-level action supports located?
- What is the spatial distribution of pollutant concentration levels among action supports that have pollutant concentrations above action level?

In many situations, the answer to the first question is known from previous studies. But if it is not known, one needs to plan the sample survey in such a way as to be reasonably sure that there are no action supports with pollutant concentrations above action level if none of the samples in the survey has a measured concentration above action level. The statistical procedures for "hot spot" detection that are used in such planning are discussed in a subsequent paragraph. The procedures for answering the other two questions were discussed earlier in the section on geostatistics. No elaborate (or simple) tests of hypotheses are required. If no samples show concentrations above action level, no remedial action is called for. If, however, a sample with proper support, which is considered reliable because of an excellent QA/QC program being in place, is obtained that has a pollutant concentration above the action level, then remedial action is called for in the neighborhood of that sample.

The problem with posing soil sampling methods and objectives in terms of population means is that the mean will depend on the area chosen. If one chooses a small area near a point source, the mean may exceed the action level; but if one increases the area so that it contains a region that is not contaminated or that is only lightly contaminated by the point source, the mean may not exceed the action limit. *Decisions on the need for remedial action should not be based on how one chooses the size of the area to be sampled, but rather on whether action supports exist that are above designated action limits.* About the only place where a comparison of means seems reasonable is in comparing the pollutant concentrations at a background (up-gradient) site with the pollutant concentrations of a site down-gradient from a suspected point source. Also, clean-up areas may be defined so that the average concentration in those units of soil must be compared with a standard.

DESIGN FOR HOT SPOT DETECTION

As stated earlier in this chapter, one of the primary questions in many environmental monitoring situations is whether there are any action supports in the study area in which the pollutant concentration exceeds the action level concentration. (We shall call an action support in which the pollutant concentration exceeds the action level a **hot spot**.) If this is a primary question in a study, then subsequent questions in the planning of the sampling design are:

- What is the probability that a sample will detect a hot spot? and
- What is the probability that a hot spot exists when no hot spot is found in the sampling?

The procedures for addressing these design problems are discussed in more detail by Gilbert (1987).

The assumptions that will be made in this discussion are the following:

(1) the hot spot is circular in horizontal cross-section;

(2) samples are taken on a square grid;

(3) the distance between grid points is much larger than the sample support diameter;

(4) there are no measurement misclassification errors (i.e., if a sample comes from a hot spot, the measured pollutant concentration in the sample will exceed action level; and, if the sample is not of a hot spot, the measured pollutant concentration will be below action level); and

(5) either the hot spot or an initial point in defining the sampling grid is randomly located within the site.

(Gilbert [1987] allows elliptical hot spots and rectangular or triangular grids in his discussion.)

Let R represent the radius of a hot spot and D be the distance between adjacent grid points where samples will be collected. The probability that a grid point will fall on a hot spot is easily obtained from a geometrical argument since at least one grid point must fall in any square of area D^2 centered at the center of the hot spot. From this concept, it follows that the probability of sampling a hot spot is given by

$$
\begin{aligned}
P(H) &= (\pi R^2) / D^2 \qquad \text{if } R \le D/2 \\
&= \{R^2[\pi - 2 \arccos (D/(2R))] + (D/4)\sqrt{(4R^2 - D^2)}\}/D^2 \qquad \text{if } D/2 < R < D\sqrt{2}/2 \\
&= 1 \quad \text{if } R \ge D\sqrt{2}/2
\end{aligned}
$$

where the angle whose cosine is $D/(2R)$ is expressed in radian measure. If the grid spacing is taken to be $D = 2R$, the probability of a hit is $\pi/4 = 0.785$, which implies that the probability that this grid spacing would not hit a hot spot if it exists is 0.215.

The second question concerning the probability that no hot spot exists (given that none was found) requires the use of a subjective probability, $P(E)$, based on historical and perhaps geophysical evidence of the existence of a hot spot on the site. Then, if $\bar{E}$ is the event that there are no hot spots at the study site and if $\bar{H}$ is the event that no hot spot is sampled in the survey, Bayes formula gives

$$
\begin{aligned}
P(E \mid \bar{H}) &= P(\bar{H} \mid E)\, P(E) / [\, P(\bar{H} \mid E)P(E) + P(\bar{H} \mid \bar{E})P(\bar{E})] \\
&= P(H \mid E)\, P(E) / [P(\bar{H} \mid E)P(E) + P(\bar{E})].
\end{aligned}
$$

For the case where $D = 2R$, it was found that $P(\bar{H} \mid E) = 0.215$, so if one is given that the chance $P(E)$ of a hot spot is thought to be 0.25 prior to the investigation, the probability of a hot spot existing if the study does not find a hot spot is

$$P(E \mid \text{no hit}) = 0.215\,(0.25) \,/\, [0.215\,(0.25) + 0.75] = 0.067.$$

Hence, the probability that no hot spot exists is $(1 - 0.067) = 0.933$.

SOME CLASSICAL STATISTICAL PROCEDURES

In this section, classical tests of hypotheses, confidence intervals, and prediction intervals based on the Student's t-distribution will be discussed. While these procedures do not apply to the three primary questions listed above concerning the existence and location of action supports above action level, they may be useful in comparing pollutant concentrations in regions up-gradient and down-gradient from a possible point source. It should also be pointed out that these procedures are only applicable to random samples (i.e., not to systematic grid samples), and great care is required in using them for anything other than simple random samples. The basic assumptions of these procedures are that the data in a sample are independent and identically distributed (with the distribution being a normal distribution) and that the measurement error variance (particularly the between-batch error variance) is a very small part of the total variance of the measurements in a sample survey of a region.

Confidence Intervals

Often one wishes to estimate the concentration of measured pollutant over an action support or over a larger subregion of a study area and to indicate the precision of the estimated concentration. The precision may be indicated by a variance, standard deviation, coefficient of variation, or confidence interval for the expected value (mean) μ of the concentration. Where statistical designs involving randomization in the selection of all sample points are employed, the analysis of variance table (see example in Chapter 9) often provides needed information for the calculation of these quantities and intervals.

The confidence interval is bounded by confidence limits which represent the bounds of the uncertainty caused by the variability of the data in the study. A two-tailed confidence interval for μ based on the assumptions stated above is of the form

$$\bar{x} - ts/\sqrt{m} \leq \mu \leq \bar{x} + ts/\sqrt{m},$$

where $\bar{x}$ is the sample mean, $s/\sqrt{m}$ is the standard error of that sample mean, m is the number of samples, and t is a value obtained from a table of the Student's t-distribution (see Appendix B) at the desired level of confidence and with the degrees of freedom associated with the estimate s. When dealing with confidence intervals, the level of confidence is a percentage but it is **not** a probability. One has a certain level of confidence in a particular statement because the procedure used to derive the statement gives correct statements that percentage of the time, but the particular statement (confidence interval) is either correct or false; it does not have a probability of being correct. The point of confusion is that the term "confidence level" is sometimes used in connection with tests of hypotheses as a probability that one will not make a Type I error in performing the test.

Consider again the example in Chapter 9 involving the analysis of variance of hypothetical data (Table 8) from a stratified random sampling design. If all the strata represent equal-area subdivisions of the study area, the logical estimate of the mean concentration over the study area is just the sample mean of all 24 measurements taken; that is, $\bar{x} = 82.3/24 = 3.43$. This sample mean could also be obtained by first averaging over each pair of subsamples and then averaging these 12 sample values. The variance of the average over the pair of subsamples is $(\sigma_A^2/2)$. When one averages the 12 samples, a new source of error is added; namely, the samples-within-strata variance, σ_B^2. Therefore, the variance of the sample mean is

$$[\sigma_B^2 + \sigma_A^2/2]/12 = (\sigma_A^2 + 2\sigma_B^2)/24.$$

The quantity, $(\sigma_A^2 + 2\sigma_B^2)$ is estimated by the mean square for samples within strata which had 8 degrees of freedom associated with it in the analysis of variance table of that prior example. The value of that mean square was 0.3199. Therefore, our estimate of the standard error of the mean, $s/\sqrt{m}$, is

$$\sqrt{0.3199} / \sqrt{24} = 0.155.$$

The table in Appendix B gives $t = 2.306$ for a two-tailed confidence interval with a 95 percent level of confidence based on 8 degrees of freedom. Hence, the 95 percent confidence interval for the mean is given by

$$3.43 - (2.306)(0.115) \leq \mu \leq 3.43 + (2.306)(0.115),$$

which is

$$3.16 \le \mu \le 3.70.$$

Prediction Intervals

Prediction intervals (see Hahn, 1969, or Guttman *et al.*, 1982) are similar to confidence intervals in appearance but are used to give an interval estimate of one future randomly chosen sample value. If that one additional sample is to be taken from stratum i, the defining two-tailed interval for the one future value, x_{if} (say), is

$$\bar{x}_i - ts\surd[(1/n)+(1/bn)] \le x_{if} \le \bar{x}_i + ts\surd[(1/n)+(1/bn)],$$

where $\bar{x}_i$ is the sample mean for stratum i. Hence, one can say for the above example that if one more sample were randomly taken from stratum 1 (which had sample mean 2.46), one would be 95 percent confident that the mean of the analyses of the two subsamples of that sample would give a value x_{if} such that

$$2.46 - (2.306)(\surd 0.3199)\surd[(1/2) + (1/6)] \le x_{if} \le 2.46 + (2.306)(\surd 0.3199)\surd[(1/2) + (1/6)],$$

which is

$$1.40 \le x_{if} \le 3.52\ .$$

One-tailed confidence and prediction intervals can be obtained using the same methods, only leaving off the bound on one side and using the "one-tailed" heading on the t-table in Appendix B.

Tests of Hypotheses

Probably the most commonly used test of hypotheses for comparison between two population means or the comparison of a population mean with some standard value (e.g., action level) is a t-test. To compare two means, μ_1 and μ_2, using data from simple random samples of the two populations, the following test statistic is employed:

$$t_s = (\bar{x}_1 - \bar{x}_2) / \{s_p\surd[(1/n_1) + (1/n_2)]\}$$

where the pooled standard deviation,

$$s_p = \surd[\{(n_1-1)s_1{}^2 + (n_2-1)s_2{}^2\}/(n_1+n_2-2)]$$

and $\bar{x}_i$, s_i^2, and n_i are the sample mean, sample variance, and sample size of sample i (i=1,2). This two-sample t-test requires one additional assumption to the ones mentioned earlier; namely, it is assumed that the population variance is the same for both populations sampled. The test is of the null (no difference) hypothesis H: $\mu_1 = \mu_2$ versus either the two-tailed alternative A: $\mu_1 \neq \mu_2$, or a one-tailed alternative such as A: $\mu_1 > \mu_2$. For the two-tailed alternative, one accepts the alternative hypothesis only if $| t_s | \geq t$, where t is the value found in the table of Appendix B and listed in the (1-α) column, for two-tailed alternatives, and in the (n_1+n_2-2) degrees of freedom (df) row. For the one-tail alternative, one accepts the alternative hypothesis only if $t_s \geq t$, where the value t is again found as before, only now use the (1-α) column for one-tailed tests.

The need to use a one-sample t-test which compares a population mean against a standard value may arise in determining whether the mean concentration of a pollutant in a study area or clean-up unit of a study area exceeds a specified action level. The test statistic for this test is

$$t_c = (\bar{x} - L)(\sqrt{n})/s,$$

where L is the action level, and n is the sample size. One- and two-tailed tests of H: $\mu = L$ versus A: $\mu \neq L$ or A: $\mu > L$, are performed in the same way as described for the two-sample tests, except now the degrees of freedom are (n-1) for the one-sample tests.

Example: A preliminary study is done in an area suspected of being contaminated with polychlorinated biphenyls (PCBs). Sixteen soil samples were collected from both the study area and from a background area through the use of simple random sampling. It was decided before sampling that a t-test of H: $\mu_S = \mu_B$ versus A: $\mu_S > \mu_B$ will be performed on the data and that the probability of making a Type I error (i.e., accepting A when H is true) will be limited to 1 percent ($\alpha = 0.01$). Table 8 lists the concentration measurements.

TABLE 8. PCB MEASUREMENTS (HYPOTHETICAL DATA)

Background Area (ppb)		Study Area (ppb)	
35.8	38.5	47.0	50.0
45.5	36.0	62.0	49.6
35.5	40.5	47.0	53.5
32.0	35.5	59.5	68.0
50.0	45.5	40.0	60.0
39.0	37.0	57.5	45.0
37.0	36.0	48.5	42.5
47.0	53.0	53.0	58.7

$\bar{x}_B = 40.23$ $s_B^2 = 36.8825$ $n_B = 16$ $CV_B = 15.1\%$

$\bar{x}_S = 52.61$ $s_S^2 = 60.2598$ $n_S = 16$ $CV_S = 14.8\%$

The test statistic is calculated as follows:

$$s_p = \sqrt{[15(36.8825 + 60.2598)/(16 + 16-2)]} = 6.97$$

$$t_s = [52.61 - 40.23]/[6.97\sqrt{(2/16)}] = 5.02$$

The critical value for t for an $\alpha = 0.01$, one-tailed t-test with 30 degrees of freedom is found in Appendix B to be 2.457. The observed value of the test statistic 5.02 is larger than the critical value, so it would be concluded that the mean concentration for PCB is larger in the study area than in the background area. A one-sided 99 percent confidence interval for $\mu_s - \mu_B$ is

$$\mu_S - \mu_B > \bar{x}_S - \bar{x}_B - ts\sqrt{[(1/n_S) + (1/n_B)]} = 6.28 \text{ ppb.}$$

One might also wish to test whether the mean concentration of the study site is above an action level of 50 ppb. Now one uses a one-tailed, one sample t-test of H: $\mu_S \leq 50$ versus A: $\mu_S > 50$. Here the maximum possible probability of making a Type I error is set at 5 percent ($\alpha = 0.05$) for illustrative purposes. The test statistic takes the value

$$t_c = (52.61 - 50.00)(\sqrt{16})/\sqrt{60.2598} = 1.34.$$

The critical value found from the table in Appendix B for a one-tailed test with $\alpha = 0.05$ and 15 degrees of freedom is 1.753. Since the value of the test statistic is less than the critical value, one does not accept the alternative hypothesis. Here one must worry whether the alternative hypothesis might have been accepted if more samples had been taken. That is, has a Type II error been committed in this case for lack of sufficient information?

In the use of one-tailed t-tests and confidence intervals such as those illustrated in the above example, one needs to worry about the assumption of normal distribution for the data and the equality of population variances. While two-tailed tests are relatively robust with respect to these assumptions, one-tailed tests are not. Unfortunately, as has been pointed out before, the underlying distribution of the population of pollutant concentrations can be quite nonnormal and also difficult to transform to normality. Further, there is no reason to expect the population variances to be equal in two different regions. To avoid this problem in one-tailed procedures, one may prefer to employ rank tests (see Lehmann, 1975) that do not require distribution assumptions.

CHAPTER 11

SAMPLE DOCUMENTATION, COLLECTION AND PREPARATION

INTRODUCTION

An important segment of a study's QA/QC plan deals with sample documentation, collection, and preparation methods. These aspects of the definitive study must be identified and appropriately applied if the specific objectives of the sampling/monitoring effort are to be met. Improperly collected and documented samples can void the entire study. As such, the final protocol must provide guidance and identify sample collection and handling methods, equipment requirements, sampling locations, documentation requirements, sample compositing requirements and methods, and the depth or depths that will be sampled.

The authors recommend that the RPM or investigators be able to estimate the components of variance or error associated with each element of the sample collection and preparation methods and procedures used from the data generated by the study. Evidence from the exploratory study pertinent to this estimation process should be taken into consideration. It is recommended that a minimum adequate documentation and sample methodology approach be selected consistent with the objectives of the study, the resources available and the designated levels of precision and confidence. It is important that criteria or procedures for determining, during and after the fact, whether or not the sample collection/preparation elements of the protocol were satisfactorily achieved. Guidance for selecting, incorporating, assessing, and interpreting sampling QA/QC data is presented in Chapter 12.

The recommendations and guidance presented in this chapter are general in nature. However, recommended detailed procedures and methods addressing and identifying documentation, soil sample collection methods, and soil sample preparation methods are presented in a number of reports including the following working protocols and guidance documents:

Documentation of EMSL-LV Contribution to Dallas Lead Study
U.S. EPA EPA-600/4-84-012 1984. Las Vegas, Nevada

Sampling for Hazardous Materials U.S. EPA OERR, Environmental Response Team. Washington, D.C.

The Environmental Survey Manual U.S. DOE Volumes 1-4 DOE/EH-0053 1989. Washington, D.C.

Refuge Contaminant Monitoring Operations Manual. Soil Sampling Reference Field Methods U.S. FWS. 1988. Prepared by USDOE/INEL/EG&G, Idaho Falls, Idaho.

Preparation of Soil Sampling Protocol: Techniques and Strategies. U.S. EPA EPA-600/4-83-020 1983. Las Vegas, Nevada

National Enforcement Investigations Center Policies and Procedures. U.S. EPA NEIC EPA-330/9-78-001-R 1986. Denver, Colorado

Refuge Contaminant Monitoring Operations Manual. Documentation Guidance Standard Operating Procedure. U.S. FWS 1989. Prepared by USDOE/ INEL/EG&G, Idaho Falls, Idaho.

DOCUMENTATION

Documentation establishes procedures and identifies written records that must be incorporated into the operating procedures for sampling/monitoring efforts. Document control procedures are required for the following three reasons:

- Enhances and facilitates sample tracking and the interpretation of sampling and analytical data.
- Standardizes data entries for input into data management systems for efficient retrieval and data manipulation.
- Identifies and establishes the authenticity of data collected for possible remedial measures.

The first parameter addresses the need to view sampling and analysis results as a function of data quality and data application. Knowledge of the circumstances under which the samples were collected, handled, preserved, transported, and analyzed will play an important role in how analytical data are used and interpreted.

The second parameter addresses the need for uniformity in data recording, as a number of sampling teams may be involved in sample collecting and data gathering. As such, a consistent, standardized documentation program is essential for developing an effective and efficient data management system. The third parameter addresses the potential for the adjudication of sampling and analysis results and the associated role that evidentiary proceedings may play in remedial measures.

Contaminant monitoring from an enforcement remedial perspective will involve information gathering procedures that are more restrictive on personnel, materials, and methods than procedures used for many ecological research and/or environmental surveys. As a result, some protocols previously used for collecting, handling, documenting, and shipping samples may fail to meet the demands required for contaminant sampling situations.

For EPA's contaminant sampling/monitoring efforts, record-keeping and documenting field activities are essential elements of a thorough investigation. A written record of all field data, samples, observations, and events provide the following:

- Ensures that all essential and required information is consistently acquired and preserved for current use and future reference.
- Assures timely, correct, and complete analysis for all parameters being requested.
- Satisfies quality assurance requirements.
- Establishes a chain-of-custody record for samples.
- Provides evidence in court proceedings.
- Provides solid basis for further sampling activities.

Maintaining standardized records enhances the usability of data necessary for decision making. Using standard forms also ensures that the same types of information will be recorded consistently. These records will document and support decisions regarding the existence and abatement of contaminant problems.

Document Control

Document control is a systematic procedure for ensuring that all sampling/monitoring program documents are properly identified and accounted for during program implementation and after program completion. Document control encompasses the following:

- serialized documents,
- document inventory and assignment record, and
- document file repository.

Presented in Table 9 are the program documents that are accountable and must be identified and included in the document control procedure. Also identified are those documents that are commonly serialized. For detailed guidance in the selection and use of appropriate documents, see U.S. EPA (1986), U.S. DOE (1987), and U.S. FWS (1988) (1989).

Because of the complexity and importance of proper document control it may be advisable to select an individual to oversee and coordinate all document control responsibilities. The size and magnitude of the sampling effort will be a determining factor in the selection of a document coordinator. This decision must be made on a case-by-case basis.

TABLE 9. ACCOUNTABLE DOCUMENTS

Document Control Identifiers and Headers	Serialized
Sampling plan	Sample identification documents
Quality assurance plan	Tags
Analytical forms	Chain-of-custody documents
Log books	
Field data records and forms	
Shipping forms	
Correspondence	
Photographs, maps, drawings, etc.	
Check-Out logs	
Litigation documents	
Final report	

A number of documents identified in Table 9 require only the proper program identifier title and header (e.g., photographs, correspondence). Others, such as project logbooks, chain-of-custody forms, field data forms, and sample identification documents, require detailed entries. The header, consisting of the program identifier (title or code), Section, Revision, Date, and page___ of ___ should be placed on the upper right-hand corner of each page of documents such as QA/QC plans and sampling/analytical protocols.

Document inventory will provide document accountability to the appropriate data users and to those who will use the data results to make decisions. For example, decisions and actions taken concerning any changes in samples/monitoring methodology or any remedial measures will be available. All documents should be cataloged, categorized, and have a unique program identifier that identifies the region, specific site and year sampling/monitoring activity was conducted.

After the sampling/monitoring program has been completed, all documents generated should be assembled and stored in a program file or repository. The RPM or his/her designee is responsible for ensuring that the collection, assembly, and inventory of all program documents are completed. This document file repository should have an index identifying all included program documents and a system that identifies the disposition of and location of all original and copied documents. This file is considered accountable; therefore, any documents leaving the repository must be signed out.

The following general guidance will apply to all documents for contaminant sampling/monitoring program:

- Entries made in logbooks, field records and forms, sample labels and tags, and chain-of-custody documents should be made only with waterproof ink and/or grease pencils. If lead pencils or other writing instruments are used, note the reason in the logbook.

- Correct errors by drawing a single line through the error and enter the correct information.

- Initial and date all corrections. (A list of names and initials should be part of the written record.)

- Enter in the logbook the location and disposition of voided documents by recording their serial number (serialized documents) and/or program header and identifier information.

- Place pre-numbered (serialized), and voided documents in the program file for accountability.

- Use only bound logbooks.

Logbooks

Logbooks are maintained to (1) record, identify and describe all pertinent sampling/monitoring activities and (2) to record quantitative information for each sample collected. Included with a contents page for easy reference, the field logbook should also address and describe team activities (e.g., activity log) sampling site descriptions and sample descriptions, including field measurement data (e.g., sample log).

Field Data Records and Forms

A number of sampling/monitoring situations may require the collection of field data that necessitates the use of specialized field data forms such as profile descriptions, core logs, and field measurements (e.g., pH, temperature). When specialized forms are used, they must be included in the Field Logbook or, if more convenient, bound in a separate Field Data Records and Forms Logbook. If a Field Data Records and Forms Logbook is required, it must have the appropriate identifier and header and be categorized by sample matrix with an appropriate table of contents page. This logbook becomes a part of the program file and must be filled out and handled as previously identified for all program documents.

Sample Labels and Tags

Sample labels and tags are required for properly identifying samples and evidence. The data obtained from samples collected for a sampling/monitoring activity may be used for remedial measures. All samples must be properly labeled and tagged.

It is recommended that physical samples be identified with a label and a tag. Both sample labels and sample tags must accompany physical samples to the analytical laboratory. However, while the sample label will be disposed of with the sample, the sample tag must be kept as a permanent record in the program files. The sample tag should be returned to the originator and/or the custodian of the program files as physical evidence of sample receipt and analysis, and may later be introduced as evidence in litigation proceedings.

Chain-of-Custody

Chain-of-Custody (COC) is mandatory in all cases that involve litigation. Chain-of-Custody records perform three functions: (a) records who has custody of a sample, (b) identifies who takes possession of a sample when it is transferred, and (c) verifies that a sample was constantly under custody between sample collection and laboratory analysis.

According to the U.S. EPA's National Enforcement Investigation Center's (NEIC) Policies and Procedures (1986), in-situ measurements can be considered and will constitute evidence. A sample collected from a site for the determination of contaminants can be considered as physical evidence.

A sample is under custody if:

- it is in your possession,
- it is in your view, after being in your possession,
- it was in your possession and you locked it up, and
- it is in a designated secure area.

To establish the integrity of samples, it is necessary to demonstrate that the samples were maintained under custody from the time they were collected in the field to the time they were analyzed in the laboratory.

The Chain-of-Custody Record form must list all transfers in the possession of samples. (See U.S. EPA (1986), U.S. EPA (1984), U.S. DOE (1987), and U.S. FWS (1989) for guidance.) Properly used, this piece of documentary evidence will attest that the sample was constantly under custody between sample collection and laboratory analysis.

While being shipped from the field to the laboratory, samples pass through the hands of postal clerks, couriers, and others who are unidentified. The samples, however, are effectively in a secure area. NEIC procedures require that a custody seal be affixed to the shipping container in such a way that, if the shipping container is properly secured and arrives at the laboratory with the custody seal intact and with adequate documentation, the integrity of the samples can be demonstrated.

SAMPLE COLLECTION

Devices for collecting samples must successfully operate in conditions such as sand, silt and clays in rocky, dry, and wet environments, surface area sampling requirements, depth requirements, and must be able to collect the required volume. In addition, the sample collection device should provide the most cost-efficient sample over the total sampling effort.

U.S. EPA (1983), U.S. EPA (1983a), U.S. EPA (1988) and U.S. FWS (1988) provide information on available soil sampling devices and their operational requirements. Soils are extremely complex and will provide investigators with a multitude of sampling situations. As a result, no single sampling method can be recommended. Sampling personnel will have to select the method that will best accommodate their sampling needs and that will satisfy the stated program objectives. Sampling devices must be carefully cleaned prior to and between each sample to avoid cross contamination. Suggested cleaning or decontamination procedures are presented in U.S. DOE (1987), U.S. FWS (1989a) and in U.S. EPA (1982) and (1984).

Frequency of Sampling

Frequency of sampling depends on program objectives, sources of pollution, pollutants of interest, transport rates, and disappearance rates (physical, chemical or biological transformations, as well as dilution or the determination of dispersion). Sampling frequency may be related to changes over time, season, or precipitation. Normally little information will be obtained on sampling frequency from the exploratory study, but in those cases where temporal changes are expected, the final study should address sampling frequency in the design and in the selection of sampling devices. It is not uncommon for many definitive studies to be conducted over a period of one year or more or through cycles of wet and dry environments.

Rapid changes expected in the concentration of pollutants in soil are normally associated with precipitation. Precipitation may influence the movement of chemical pollutants downward and aids in decomposition. Sampling frequency associated with either major rainfall events or with accumulated amounts of rainfall can often provide valuable information on changes that are occurring.

Monitoring studies are often designed to measure the effects of some remedial measure on the site. Trends are important in these cases. The frequency of sampling should be designed to measure changes, e.g., efficiency of remedial measures. One approach used successfully has been to provide intensive initial sampling early, then decrease sampling frequency as the levels begin to drop. One recommended procedure would be to sample monthly for the first year, quarterly for the second year, semiannually for the next two to three years, then annually thereafter.

Evaluation of the trend of the data should allow the RPM to determine when the sampling frequency can be reduced or halted completely. Monthly sampling may provide the needed data for performing statistical tests and for determining the yearly variation.

Samples collected for evaluating trends can usually be obtained on some subset of the initial year's sampling. The major focus is mainly on the highly contaminated and on the immediately adjacent areas. The investigator is primarily interested in detecting changes in these adjacent areas in order to provide early warning of the efficacy of remedial measures.

SAMPLE PREPARATION

Sample preparation encompasses all physical handling of sample(s) following the actual collection. This includes, but is not limited to the following:

- transfer from the collecting device,
- sieving/mixing procedures,
- drying methods and procedures,
- selecting and using containers,

- preservation,
- archiving (storage), and
- transportation and shipment.

It is inappropriate to initiate a sampling effort without first becoming familiar with sample preparation requirements. For example, it is not recommended to dry and sieve samples that are collected for the determination of volatile contaminants. Collecting samples that cannot be suitably analyzed will not yield high quality decision-making data, thereby compromising achievement of the sampling/monitoring objectives.

In addition to the protocols and guidance documents previously identified, recommended soil sample preparation methods for different contaminant analyses are presented in U.S. EPA (1986a), U.S. EPA (1989), OSU (1971), and by Peterson and Calvin (1965).

Sieving/Mixing

Note: Sieving and mixing can only be carried out on soils containing pollutants with little or no tendency to vaporize. This requires that the techniques discussed below not be used for volatile pollutants.

Analytical methods that are used by the U.S. EPA to analyze soils have been validated with a prescribed sample volume and a specified particle size. As such, for the best analytical results, the analyst must be provided with a sample that is commensurate with the analytical requirements. The responsibility for providing the appropriate sample for analysis lies with sampling personnel. This responsibility also includes the requirement to prepare (e.g., mix and sieve) soil samples in a prescribed manner to provide a representative sample from the total soil material collected. For example, when single- or multiple-sample cores are collected for compositing, it is recommended that the samples be prepared before they are shipped to the analytical laboratory.

Soil sieving/mixing sample preparation methods must satisfy the following requirements:

- provide the specified amount of material,
- provide a representative aliquot of the total sample collected, and
- provide an adequate and appropriate sample to enable analyses for the required contaminants (e.g., volatiles, semivolatiles, metals).

It is extremely rare to collect soil that does not contain non-soil components (e.g., rocks, non-mineral material). Also, in many soils organic matter is commonly found and is an integral part of the soil matrix. Both the non-soil components and the organic matter may play an important role in the interpretation of the analytical data (e.g., the non-soil components may be a source of contamination to the soil matrix).

The potential for errors being introduced in the sample sieving and mixing procedures is high, especially involving discarded non-soil or non-sieved material, as well as possible physical and/or chemical losses during any grinding or drying operation.. Decisions concerning the non-soil fraction may be made on the basis of data obtained from an exploratory study. Available data may indicate that significant contamination is in the discarded portion. If so, it is recommended that the discarded portion from ten percent of the samples collected from areas having the highest concentrations be analyzed. An estimate can then be made of the total amount of contamination being discarded by multiplying the measured concentration in the discarded material by the total amount of the discarded material. Assuming that this amount is uniformly distributed through the soil sample remaining after non-soil and non-sieved materials have been discarded, one can then calculate an estimated value for the potential soil sample total concentration, if none of the contamination had been discarded. Comparison of this potential concentration to the actual measured concentration will enable an estimate of the possible error related to discarded contamination.

If the error estimated by this process exceeds acceptable limits specified in the QA/QC plan, it might be necessary to modify sample preparation procedures for the definitive study. One might consider a sample sieving and mixing procedure in which the entire collected sample (soil and non-soil materials) is extracted in the analytical laboratory. The analytical results could then be reported as amounts of contaminant per gram of mixed material. At present there is no acceptable method for proceeding in cases such as these. One problem is the lack of standard reference materials for determining and measuring errors in extraction efficiency. One solution may be to try different methods of extraction and compare the results. The final interpretation of the data must then take into consideration these estimated errors.

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Sample Containers

The current EPA recommended container, preservation and holding time requirements for specific contaminants is shown in Table 10. Recommended sample volumes are presented in U.S. EPA (1983a), U.S. DOE (1987), and U.S. FWS (1989).

It is recommended that sample containers be obtained from a commercial source that provides containers cleaned to EPA-approved specifications. The cleaning procedures used should be EPA approved. Also, sampling personnel should check current container and sample volume recommendations as improvements in containers, materials used in their construction, holding time requirements, preservation procedures, and analytical protocols are consistently being updated and improved.

Archiving

A number of sampling/monitoring circumstances may require the archiving or storing of collected samples or portions of collected samples that have been submitted for analysis. For example, the design of a monitoring program may require that a large number of samples be collected. If there is uncertainty as to the definitive identity of a contaminant(s) or cost of analysis is a concern, an alternative for analyzing all of the samples collected is to select only a small number of them for analysis. Following the analysis and data assessment of these initial samples, a decision to analyze additional samples can be made. Additional reasons for archiving samples is to provide a "back-up" if a sample is lost or spilled, and/or when additional analysis is necessary for validating an unexpected or unusual (exceedingly high or low) result. When samples are being archived, the samples should be stored in containers and under the preservation requirements presented on Table 10. If samples are stored for a period longer

TABLE 10. SAMPLING CONTAINERS, PRESERVATION REQUIREMENTS, AND HOLDING TIMES FOR SOIL SAMPLES

Contaminant	Container	Preservation	Holding Time
Acidity	P,G	Cool, 4°C	14 days
Alkalinity	P,G	Cool, 4°C	14 days
Ammonia	P,G	Cool, 4°C	28 days
Sulfate	P,G	Cool, 4°C	28 days
Sulfide	P,G	Cool, 4°C	28 days
Sulfite	P,G	Cool, 4°C	48 hours
Nitrate	P,G	Cool, 4°C	48 hours
Nitrate-Nitrite	P,G	Cool, 4°C	28 days
Nitrite	P,G	Cool, 4°C	48 hours
Oil and Grease	G	Cool, 4°C	28 days
Organic Carbon	P,G	Cool, 4°C	28 days
Metals			
Chromium VI	P,G	Cool, 4°C	48 hours
Mercury	P,G	Cool, 4°C	28 days
Metals except above	P,G	Cool, 4°C	6 months
Cyanide	P,G	Cool, 4°C	28 days
Organic Compounds			
Extractibles (including phthalates, nitrosamines organochlorine pesticides, PCB's nitroaromatics, isophorone, polynuclear aromatic hydrocarbons, haloethers, chlorinated hydrocarbons and TCDD)	G, teflon-lined cap	Cool, 4°C	7 days (until extraction) 30 days (after extraction)
Extractables (phenols)	G, teflon-lined cap	Cool, 4°C	7 days (until extraction) 30 days (after extraction)
Purgables (halocarbons and aromatics)	G, teflon-lined septum	Cool, 4°C	14 days

(continued)

TABLE 10. (Continued)

Contaminant	Container	Preservation	Holding Time
Purgables (acrolein and acrylonitrate	G, teflon-lined septum	Cool, 4°C	3 days
Orthophosphate	P,G	Cool, 4°C	48 hours
Pesticides	G, teflon-lined cap	Cool, 4°C	7 days (until extraction) 30 days (after extraction)
Phenols	G	Cool, 4°C	28 days
Phosphorus	G	Cool, 4°C	48 hours
Phosphorus, total	P,G	Cool, 4°C	28 days
Chlorinated organic compounds	G, teflon-lined cap	Cool, 4°C	7 days (until extraction) 30 days (after extraction)
Polyethylene (P) or Glass (G)			

P = polyethylene
G = glass

Sample preservation should be performed immediately upon sample collection. For composite samples, each aliquot should be preserved at the time of collection. When impossible to preserve each aliquot, then samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still considered valid. Samples may be held for longer periods only if the analytical laboratory has data on file to show that the specific types of samples under study are stable for the longer time.

For additional information see U.S. EPA (1983a).

than the stated holding time, a decision concerning the utility of the data obtained from their analysis must be made. This decision would have to be made by the data user on a case-by-case basis as a function of the intended use of that particular data and should be documented as necessary.

Sample Bank

For sampling studies that require a large number of samples and/or extensive pre-analytical sample preparation, a sample bank may be advantageous. The sample bank is the element that operates between the field sampling effort and the analytical laboratory. It is established to handle the distribution and preparation of samples for large sampling efforts (U.S. EPA, 1980). However, for smaller studies the sample bank's responsibilities are often incorporated into the responsibilities of the field sampling team or the analytical laboratory.

The following sample bank responsibilities and procedures have been used successfully on a number of soil monitoring studies (U.S. EPA 1982, 1984, 1989).

A. Issuing Supplies:

(1) The sample bank issues, as required, sample containers, sample collection tags, chain-of-custody forms, and site description forms to the sampling teams. Sample collection tags and chain-of-custody forms are normally accountable documents; the sample bank will log the forms by numerical lot identifying the team and/or the individual responsible for the temporary custody of these documents.

(2) The sample bank may be required to store sampling equipment in a suitable environment. If sampling equipment is stored at the sample bank, issuing this equipment to the sampling teams as required will be necessary.

B. Record Keeping

(1) Custodian for all records pertaining to the sampling, sample preparation as required, and shipment of soil samples to analytical laboratories.

(2) Responsibility for record filing and storing, for storing and preparation of soil samples, and for dispensing containers, sampling equipment and all custody documents such as chain-of-custody forms and sample

collection and analytical tags, as required.

(3) Responsibility for updating and maintaining the project's master log book, auditing the records as required, generating QC samples (e.g., sample bank blanks, splits, etc.), accepting QA/QC samples for inclusion into the analytical scheme, and for scheduling the collection of field sample blanks.

(4) Responsibility for completing, as required, analysis data reporting forms and for assuring that all chain-of custody requirements pertaining to all field sampling, shipping and sample bank operations are adhered to.

(5) All unused accountable documents as shown in Table 10 must be returned to the sample bank on a daily basis. However, depending upon circumstances, such as a sampling team's schedule and route, accountable documents may be retained by the sampling team leader. The sample bank supervisor, however, must be aware of the situation.

Preparation of soil samples for analysis normally requires sample bank personnel to dry, sieve, mix and aliquot samples appropriately. The preparation procedures selected must be identified in the protocol and adequately address the contaminant(s) to be measured and the analytical requirements.

QUALITY ASSURANCE ASPECTS

QA/QC procedures of the sample documentation/collection effort must identify and determine the magnitude of errors associated with characterizing soil contamination introduced through the sample collection effort. Audits (Chapter 13) are an effective tool for insuring that sampling is being done as specified. Factors most likely to influence the magnitude of the sample collection error are collection and preparation methods, and frequency of sampling. Perhaps the most important of these are preparation methods and frequency of sampling.

The tools and equipment used for collecting and preparing soil samples themselves are not likely to be sources of error. Errors will most likely occur in the inconsistent use of these devices. Proper replication, decontamination and appropriate QC sample selection, analysis, and assessment will insure that the precision of the procedure(s) meets the QA/QC objectives and thence the DQOs.

CHAPTER 12

ANALYSIS AND INTERPRETATION OF QA/QC DATA

INTRODUCTION

One goal in the analysis and interpretation of data is to show how all aspects of QA/QC for a soil monitoring study combine to give an overall level of precision and confidence for the data resulting from the study. Another goal may be to determine whether all QA/QC procedures used were necessary and adequate and should definitely be incorporated into future studies of the same type. This entire evaluation must be closely linked to the objectives, and specifically, to the data quality objectives of the study. In summary, the important questions to be answered are: "What is the quality of the data (maximum accuracy attainable)?" and "Could the same objective have been achieved through an improved QA/QC design which may have required fewer resources?"

PRESENTATION OF DATA SUMMARIES

It is desirable to provide summarized tables of validated QA/QC data in the final report. For example, QA/QC data validation procedures used in a number of soil sampling studies reported by Brown and Black (1983) included validation of sample data sets by checking and assessing the accompanying QA/QC data. The criteria for QA/QC samples and procedures used to validate all data included:

Samples and Procedures		Example Criteria
1.	Reagent Blanks*	Concentrations had to be less than 0.25 μg/ml.
2.	Calibration Check* Standards	Recovery must be between 95% and 105% of the known value for either the first analysis or the first re-check analysis.
3.	Laboratory Control* Standards	Recovery must be between 90% and 110% of the known value for either the first analysis or the first re-check analysis.

*Applies to analysis of soils for lead

One of the studies discussed by Brown and Black (1983) involved lead-contaminated soils. The results of the QC analyses for this soil monitoring study were presented as follows:

QC Sample	No.	Mean	s
Calibration Check Standard	150	101.5%	2.6%
Laboratory Control Standard	147	101.2%	4.1%
Field Blank (μg ml^{-1})	76	<0.25	
Sample Bank Blank (μg ml^{-1})	77	<0.25	
Reagent Blank (μg ml^{-1})	148	<0.25	
Re-extraction Analysis	17	1.7%	1.4%
Total Recoverable	144	99.8%	8.0%
Split Extract (CV)*	147	0.0089	0.0079
Spiked Extract	147	99.4%	5.0%
Spiked Sample	147	100.4%	5.1%
Duplicate Aliquot (CV)	134	0.053	0.047
Duplicate Sample (CV)	129	0.189	0.168
Triplicate Analysis (CV)	220	0.144	0.128

$$^{*}CV = \frac{s}{Mean}$$

From data summarized in this fashion, it is possible to determine the adequacy of the QAPP in insuring the achievement of the assigned DQOs.

It is required that the QA/QC plan document and insure that all data collected, whether used for research or for monitoring purposes, be scientifically valid, defensible and of known precision and accuracy. The described presentation of QC data, though designed for analysis of lead in soil, can be used as a guide for other sampling and data analysis protocols and/or QA/QC plans.

Presentation of QA/QC data allows readers to verify conclusions drawn as to the reliability of the data. Such an approach also contributes to the building of a body of QA/QC and monitoring experimental data in the literature which allow comparisons to be made between and among studies. Procedures used to validate the individual data points should be presented, and where some points are discarded, arguments should be presented to support these decisions.

PRESENTATION OF RESULTS AND CONCLUSIONS

Special emphasis should be placed on how overall levels of precision and confidence were derived from the data. Great care must be exercised to insure that, in determining results and conclusions, assumptions are not made which were not part of the study design and which cannot be tested by data derived from the study. If portions of the study results are ambiguous and supportable conclusions cannot be drawn with regard to the total reliability of the data, that situation must be clearly stated. In that event it is desirable to include recommendations for conducting an improved study in such a way as to clarify the observed ambiguities.

QUALITY ASSURANCE ASPECTS

The adequacy of all aspects of the QA/QC plan should be examined in detail with emphasis on defining an appropriate minimum adequate plan for future studies. Some aspects of the plan actually used may have been too restrictive, while others may not have been restrictive enough. Appropriate analyses and interpretation of the data should identify the actual situation.

Future soil monitoring studies should have checks and balances built into the QA/QC plan which will identify early in the study whether the plan is adequate and, if necessary, allow for corrective action to be taken before the study continues. This is one of the major advantages of conducting an exploratory study along the lines outlined in this report. If there are problems with the QA/QC plan, they will often be identified in the exploratory study and be corrected before major resources are expended.

There is insufficient knowledge dealing with soil monitoring studies to state with confidence which components of the QA/QC plan will be generally applicable to all soil monitoring studies and which components should vary depending on site-specific factors. As experience is gained, it may be possible to provide more adequate guidance on this subject. In the meantime, it is recommended that the best approach is to assume that important factors of QA/QC plans are site-specific, and to conduct an appropriate exploratory study at each new study site to verify that various aspects of the QA/QC plan are adequate to meet program objectives prior to proceeding with the final definitive study.

CHAPTER 13

SYSTEM AUDITS AND TRAINING

INTRODUCTION

An adequate soil sampling quality assurance program ensures that the quality of the final product meets required standards. Audits are an integral part of the quality assurance process and are vital for assuring that program procedures are being implemented. They are performed to document the implementation of the quality assurance program plan, quality assurance project plan and/or associated operational protocols.

Three types of audits are commonly used to determine adequacy of the analytical measurement system, adequacy of the data collection system, completeness of the documentation of data collection activities, and document if required data collection and data quality objectives are being met. These audits are commonly referred to as System Audits, Performance Audits, and Data Quality Audits.

- System Audits are qualitative on-site field audits that evaluate the technical aspects of field operations (e.g., sampling methods) against the requirements of approved QA plans and protocols. System audit reports note problems and recommend or allow corrective actions to be taken to protect the validity of collected data.

- Data Quality Audits are evaluations of the documentation associated with data quality indicators of measurement data to verify that the generated data are of known and documented quality. This is an important part of the validation of data packages showing that the methods and Standard Operating Procedures (SOPs) designated in the QA plans were followed and that the resulting data set is a functional part of satisfying the established DQOs. The results are vital to decisions regarding the legal defensibility of the data should it be challenged in litigation.

- Performance Audits are generally based on Performance Evaluation (PE) samples. Samples having known concentrations may be tested as unknowns in the laboratory or a sample may be analyzed for the presence of certain compounds. Performance audits are used to determine objectively whether an analytical measurement system is operating within established control limits at the time of the audit. The performance of personnel and instrumentation are tested by the degree of accuracy obtained.

Standard Operating Procedures to assist auditors in addressing critical program elements and preparing for on-site audits are presented in U.S. EPA (1985). The recommended initial phase for conducting an audit is the preparation of a program specific checklist. Examples of audit checklists and laboratory evaluations are presented in U.S. EPA (1984a, b, 1989), and for numerous sampling effects conducted for the environmental Survey Program (U.S. DOE 1987). A discussion with the RPM concerning the current status of the project and the identity of any problems encountered is suggested before conducting on-site field audits.

Audits, in most part, are conducted by appropriate elements of agencies or organizations having cognizance over a monitoring project. However, audits can be conducted by independent or third party organizations. The frequency of auditing should be determined by the RPM or project officer. Juran *et al.*, (1979), states that, "the activities subject to audit should include any that affect quality regardless of the internal organizational location." For illustrative purposes, important factors that are addressed in a systems audit will be discussed. Definitive procedures for conducting audits of analytical measurement systems are presented in (U.S. EPA, 1984a, 1989).

Specifically system audits:

- verify that sampling methodology is being performed in accordance with program requirements,
- check on the use of appropriate field QA/QC measures,
- check methods of sample handling, i.e., packaging, labeling, preserving, transporting, and archiving in accordance with program requirements,
- check program documentation, i.e., records (site description, chain-of-custody collection and analytical tags, field and sample bank log books and field work sheets),
- recommend corrective action if a problem is identified,
- assess personnel experience and qualifications if required,
- follow-up on any corrective action previously mandated,
- provide on-site debriefings for sampling team and sample bank personnel, and
- provide a written evaluation of the sampling and sample bank program.

Components of a systems audit may include sample bank operations and field operations.

SAMPLE BANK AUDIT

The primary objective is to determine the status of all Sample Bank documentation and archived samples. Emphasis is placed on:

- verifying that the documentation is in order and sufficient to establish the disposition of any sample collected,
- determining any discrepancies that currently exist and initiating corrective action as appropriate,
- verifying that the recording and documentation of QA/QC measures (blanks, duplicate spikes, blinds) is in accordance with the QA/QC plan, and
- establishing procedures for final disposition and mechanics of transfer of all Sample Bank holdings upon termination of the operation.

An initial step is to inventory the Sample Bank records and archived samples. The records that must be inspected are:

- Chain-of-custody forms, including
    - Field forms and
    - Analysis forms;
- Sample tags, including
    - Field tags and
    - Analysis tags;
- Analysis forms, including
    - Individual samples and
    - Batch sheets;
- Shipment forms;

- Logbooks, including
  - Soils and
  - Daily log.

The operational procedures inspected include:

- Preparation Procedures (sample bank or analytical laboratory),
  - Preservation,
  - Drying (if used),
  - Sieving,
  - Mixing,
  - Packaging, and
  - Shipping.
- Housekeeping,
  - Safety,
  - Decontamination, and
  - Evaluation of Swipe Samples;
- Security,
  - Forms (documents),
  - Samples; and
- Storage,
  - Sampling equipment, and
  - Archived samples (when appropriate).

Check that required documentation has been maintained in an orderly fashion, that each of the recorded items is properly categorized, and cross-checking can be easily performed. In addition, ensure that data recording conforms to approved documentation procedures.

Check archived samples. Verify that appropriate samples exist for each entry in the logbook. Review sample bank logbooks for complete sample information. In addition, checks for the identification and documentation of split and duplicate samples, and field and Sample

Bank rinsate samples must be performed. Detailed sample bank procedures are presented in U.S. EPA (1982, 1984, and 1989).

FIELD AUDITS

The primary objective is to determine the status of sampling operations. Emphasis is placed on:

- verifying that operational aspects and procedures are in accordance with the protocols and QA/QC plan,
- verifying the collection of all samples including duplicates, rinsates, and blanks,
- verifying that documentation is in order and sufficient to establish the collection location of any sample collected,
- determining discrepancies that exist and initiating corrective action as appropriate, and
- collecting independent samples.

Records inspected include:

a. chain-of-custody forms,
b. sample tags,
c. site description forms, and
d. log books.

The operational procedures inspected include:

- sampling procedures,
    - equipment,
    - techniques,
    - decontamination,
    - collection of duplicate and field blank samples,

- security,
- sample storage and transportation,
- containers
- contaminated waste storage and disposal, and
- site Description Form entries.

TRAINING

The project officer is responsible for determining that all members of his team have adequate training and experience to carry out satisfactorily their assigned missions and functions. Until a field sampling team has worked together long enough for the project leader to have verified this, it is good practice, in addition to any classroom training or experience, to conduct comprehensive briefing sessions for all involved parties. During these sessions, all aspects of the sampling protocol, including the QA/QC plan, are presented and discussed in detail. Sufficient field training exercises should follow the briefing sessions until each team member can demonstrate successfully that he can perform his job well and without delay.

In summary, the sampling effort must include classroom and field training programs that provide detailed instruction and practical experience to personnel in sample collection techniques and procedures, labeling, preservation, documentation, transport, and sample bank operational procedures. Also, any specialized training, such as field measurement procedures and documentation, should be completed by all personnel prior to their involvement in the conduction of any audits.

GLOSSARY

Absorption	The penetration of a substance into or through another.
Accuracy	Measures the bias in a measurement system; it is difficult to measure for the entire data collection activity. Sources of error are the sampling process, field contamination, preservation, handling, sample matrix, sample preparation, and analysis techniques. Sampling accuracy may be assessed by evaluating the results of field/trip blanks, analytical accuracy may be assessed through use of known and unknown QC samples and matrix spikes.
Anion	A negatively charged ion.
Background Level	Amount of pollutants present in the ambient soil due to natural sources.

Bulk density	The mass of dry soil per unit bulk volume, determined before drying to a constant weight at 105°C.
Calibration Check Standard	A standard material to check instrument calibration.
Cation	A positively charged ion.
Cation-exchange	The total of exchangeable cations that a soil can absorb, expressed either in milliequivalents per gram or in milliequivalents per 100 grams of soil.
Comparability	A qualitative parameter expressing the confidence with which one data set can be compared with another. Sample data should be comparable with other measurement data for similar samples and sample conditions. This goal is achieved through using standard techniques to collect and analyze representative samples and reporting analytical results in appropriate units. Comparability is limited to the other PARCC parameters because only when precision and accuracy are known can data sets be compared with confidence.

Completeness	Defined as the percentage of measurements made which are judged to be valid measurements. The completeness goal is essentially the same for all data uses: that a sufficient amount of valid data be generated. It is important that critical samples are identified and plans made to achieve valid data for them.
Data Quality Objectives (DQOs)	Qualitative and quantitative statements which specify the quality of the data required to support Agency decisions during remedial response activities. DQOs are determined based on the end use of the data to be collected.
Duplicate Sample	An additional sample taken near the field sample co-located to determine total within-batch measurement error variance.
External Laboratory Audit Sample	A sample of well-characterized soil that is sent directly to the laboratory for analysis. The analyte concentrations are unknown to the laboratory. This type of sample is used to estimate laboratory bias and laboratory

	batch to batch variability. It may also be used for external quality control of the laboratory.
Field Audit Sample	A sample of well-characterized soil that is taken into the field with the sampling crew, sent through the sample bank to the laboratory with the field samples to detect bias in the entire measurement process and to determine batch-to-batch variability.
Field Blank	A sample container filled with distilled, deionized water, exposed during sampling and then analyzed to detect accidental or incidental contamination.
Field Rinsate	A blank (last rinse using distilled deionized water) passed over the sampling apparatus after cleaning, to check for residual contamination.
Heavy Metals	Metals having a specific gravity of 5.0 or over.

Internal Laboratory Audit Sample	A sample of well-characterized soil whose analyte concentrations are known to the laboratory to be used for internal laboratory quality control.
Laboratory Control Standard	A sample of a soil standard carried through the analytical procedure to determine overall method bias.
Matrix	The predominant material of which the sample to be analyzed is composed.
PARCC	Precision, accuracy, representativeness, completeness, and comparability parameters.
Precision	Measures the reproducibility of measurements under a given set of conditions. Specifically, it is a quantitative measure of the variability of a group of measurements compared to their average value. Precision is

usually stated in terms of standard deviation, but other estimates such as the coefficient of variation (relative standard deviation), range (maximum value minus minimum value), and relative range are common.

Reagent Blank	A DDI water sample analyzed as a routine check for reagent contamination.
Re-extraction	A re-extraction of the residue from the first extraction to determine extraction efficiency.
Remedial Project Manager (RPM)	Manages remedial activities at assigned regional sites. Accountable for the technical quality, schedule, and cost of work.
Representativess	Expresses the degree to which sample data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Representativeness is a

qualitative parameter which is most concerned with the proper design of the sampling program. The representativeness criterion is best satisfied by making certain that sampling locations are selected properly and a sufficient number of samples are collected.

Sample Bank Rinsate	A sample (last rinse using distilled, deionized water) passed through the sample preparation apparatus, after cleaning, to check for residual contamination.
Semivolatiles	A group of organic compounds consisting of base/neutrals, acids, and pesticides that are identified in and analyzed by Method 625 in 40 CRF Part 136.
Soil classification	The systematic arrangement of soils into groups or categories on the basis of their characteristics.
Soil Profile	A vertical section of the soil from the surface through all its horizons, including C horizons.

Spiked Extract	A separate aliquot of extract that is spiked to check for extract matrix effects on the recovery of known added analytes.
Spiked Sample	A separate aliquot of a soil sample spiked with an appropriate standard reference material to check for soil and extract matrix effects on recovery.
Split Extract	An additional aliquot of the extract which is analyzed to check injection and instrument reproducibility.
Total Recoverable	A second aliquot of a sample digested by a more rigorous method to check the efficacy of the protocol method.
Triplicate Samples (Splits)	The prepared sample is split into three portions to provide blind duplicates for the analytical laboratory and a third replicate for the referee laboratory to determine interlaboratory precision.

Volatile	Solids or liquids which are relatively unstable at standard temperature and pressure and undergo spontaneous phase change to a gaseous state.

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APPENDIX A

APPLICATION OF SOIL MONITORING DATA TO AN EXPOSURE AND RISK ASSESSMENT STUDY

One of the possible purposes for soil monitoring is to provide data for input into exposure and risk assessment studies. The risk assessment study conducted by the Centers for Disease Control (CDC) to estimate an allowable concentration of 2,3,7,8-tetrachlorodibenzodioxin (TCDD) in residential soil provides an instructive example. Prior to presenting the example, however, a brief introduction to the general subject of risk assessment will be given.

Risk assessment as defined by the World Health Organization (WHO) is composed of three different elements:

- risk identification,
- risk estimation, and
- risk evaluation or management.

Risk identification involves the accumulation of sufficient evidence to warrant identifying the presence of a specific pollutant in the environment, at a defined concentration and averaging time, as possibly being an unacceptable risk to man or the environment. A formal risk identification on the basis of a qualitative value judgement requires further study to determine whether the risk is or is not acceptable.

Risk estimation is the process whereby a risk which has been identified is quantified in terms of developing estimates of the numbers of people, for example, who would suffer adverse health effects as a result of exposure to defined levels of the pollutant(s) of concern. Risk estimation requires the availability of both applicable exposure-response relationships for the adverse effect of concern in the exposed population and existing exposure distributions in the appropriate population(s). By comparing exposure distributions to exposure-response relationships, it is possible to predict the expected number of adverse effects in the exposed population(s).

Risk evaluation (or management) is the process whereby responsible public officials come to a value judgment decision as to what risk is acceptable. Social, economic, political, and health considerations generally are involved in this important decision. If the present risk as estimated in the previous step is deemed unacceptable, it is imperative that prompt action be initiated to reduce the risk to acceptable levels.

The risk assessment process is often easier to define than it is to perform. For example, the risk estimation process assumes the availability of applicable exposure-response relationships. Let us briefly examine how such relationships are developed. Figure A-1 depicts the elements of toxicologic studies designed to assess adverse effects related to exposure to environmental pollutants. Such studies are usually the basis for exposure-response relationships in humans. Note that there are many different possible testing systems, there are several possible exposure routes, the form and levels of pollutant may vary over wide limits, and there is almost an unending list of possible adverse effect end points.

Studies where different combinations of the toxicologic study elements have been examined comprehensively exist for only a very small number of substances. The situation is additionally complicated by the fact that results from experimental animals, usually at high exposures, must be extrapolated to humans, usually at much lower exposures. Also, combination effects resulting from the presence of more than one pollutant at a time in the real world are usually not assessed.

Due to variations in sensitivity, not all humans respond the same way to the same exposures. Figure A-2 shows a generalized spectrum of human responses to an environmental pollutant. Generally in the United States, an increased body burden and physiological or biochemical changes of uncertain significance are not considered to be adverse health effects.

Figure A-3 shows the possible routes of entry of pollutants to man from the generating source(s). In the development of exposure distributions to man, significant exposures via all routes of entry must be assessed. When exposures via more than one route are important, it is necessary to estimate the total exposure by appropriately summing all contributions.

Figure A-4 presents a total exposure model showing how media measurements (shown on the top line in the slide) may be converted to exposures. At the present time, the ability to quantify human exposures via skin absorption for many pollutants is not considered adequate. More research is needed here.

Testing System	Exposure Route	Form and Levels of Pollutant	Adverse Effect End Point
Microbes • Tissue cultures • Mice • Rats • Hamsters Guinea pigs • etc.	Ingestion • Inhalation • Skin absorption • Intravenous injection • Subcutaneous injection	Physical form • Chemical form • Exposure levels • Averaging times • Time dependence of exposure levels and exposure delivery	Mortality • Disease of: lungs heart kidney liver nervous system etc. • Cancer • Reduction of resistance to disease • Birth defects • Mutations • Growth rate effects • Reproductive effects • Behavioral effects • Etc.

Figure A-1. Elements of toxicologic studies to assess adverse effects related to exposure to environmental pollutants.

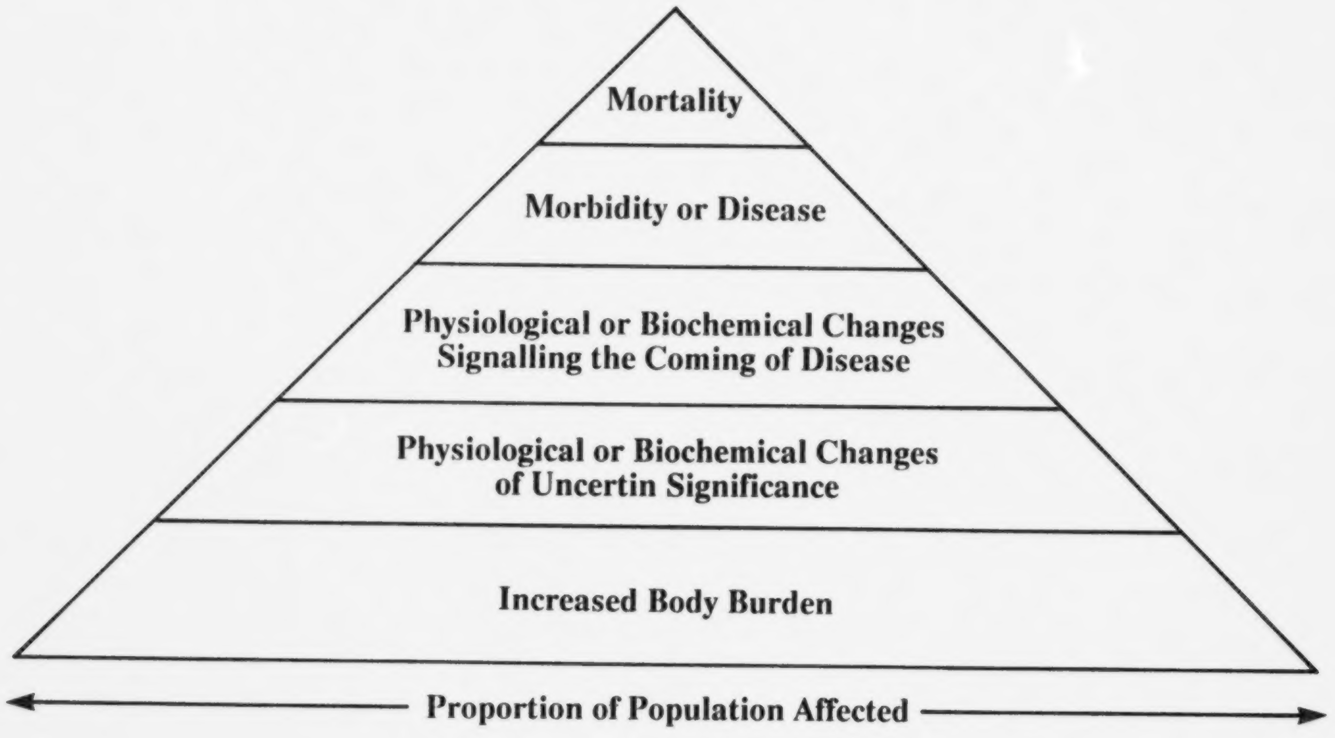

Mortality
Morbidity or Disease
Physiological or Biochemical Changes Signalling the Coming of Disease
Physiological or Biochemical Changes of Uncertin Significance
Increased Body Burden
Proportion of Population Affected

Figure A-2. Generalized Spectrum of Human Responses to an Environmental Pollutant

Figure A-5 shows the relationship of exposure to risk estimation. Note that it may be possible to infer some information about total exposure from the effects of increased body burden, or physiological and biochemical changes of uncertain significance.

Figure A-6 gives three hypothetical general classes of exposure-response relationships. Curve I shows the situation where there is no threshold of exposure which must be exceeded prior to observation of some effects. Many feel that this is the appropriate class to use for cancer-causing pollutants. Curve II shows that a threshold of exposure must be exceeded prior to observation of effects. Curve III depicts a situation where there are some effects at zero exposure. This shows that the pollutant of concern is not the only cause or contributor to the adverse effect being measured. Generally, experimental points used to define exposure-response relationships are for high exposures, and the shape and location of the curve near zero exposure is unknown. Note that if the curve is really of Class II, but is assumed to be of Class I, extrapolation of an experimental point at B through zero would seriously overestimate the effects of low exposures.

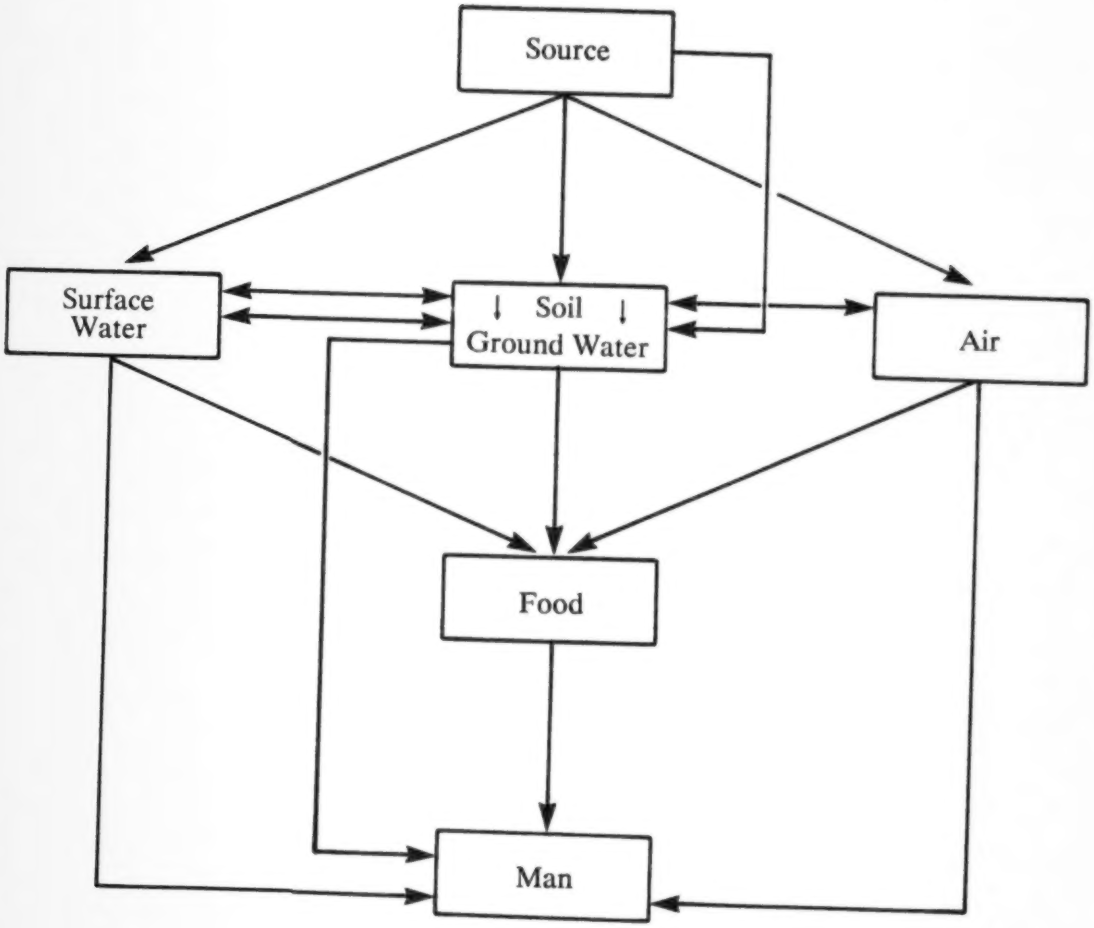

Source
Surface Water
Soil
Ground Water
Air
Food
Man

Figure A-3. Possible exposure pathways from a source of environmental pollution to man.

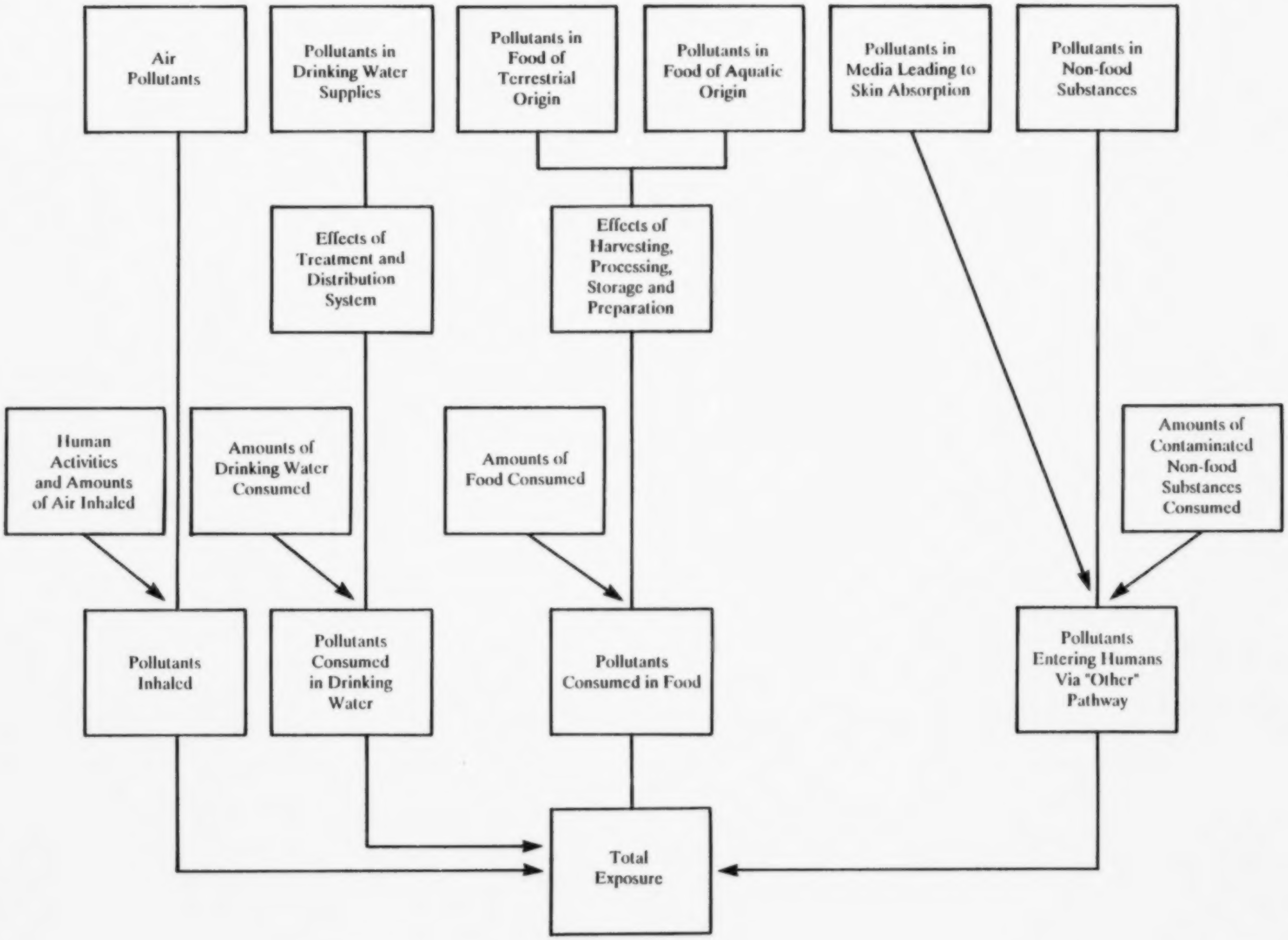

Air Pollutants
Pollutants in Drinking Water Supplies
Pollutants in Food of Terrestrial Origin
Pollutants in Food of Aquatic Origin
Pollutants in Media Leading to Skin Absorption
Pollutants in Non-food Substances
Effects of Treatment and Distribution System
Effects of Harvesting, Processing, Storage and Preparation
Human Activities and Amounts of Air Inhaled
Amounts of Drinking Water Consumed
Amounts of Food Consumed
Amounts of Contaminated Non-food Substances Consumed
Pollutants Inhaled
Pollutants Consumed in Drinking Water
Pollutants Consumed in Food
Pollutants Entering Humans Via "Other" Pathway
Total Exposure

Figure A-4. General model for converting environmental pollutant measurements in various media into estimated total exposure to humans.

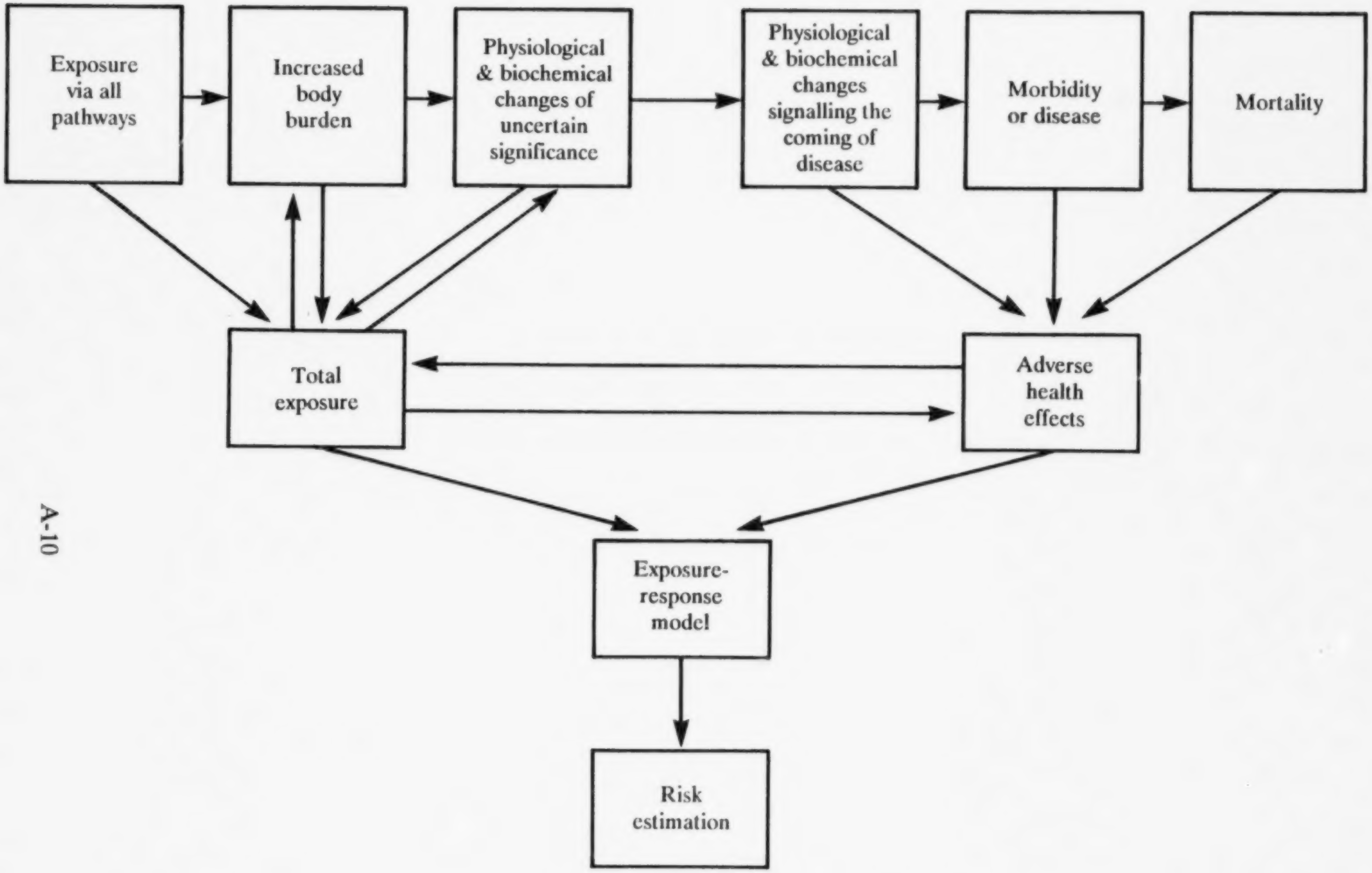

Exposure via all pathways
Increased body burden
Physiological & biochemical changes of uncertain significance
Physiological & biochemical changes signalling the coming of disease
Morbidity or disease
Mortality
Total exposure
Adverse health effects
Exposure-response model
Risk estimation

Figure A-5. Relationship of total human exposure to possible effects and to risk estimation.

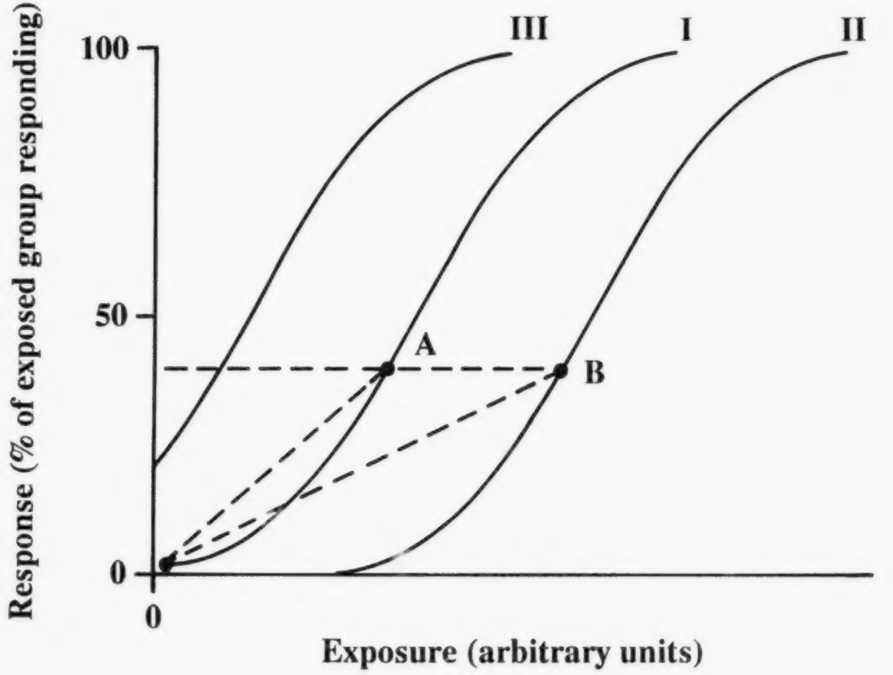


Figure A-6. Three hypothetical general classes of exposure-response relationships.

Figure A-7 depicts exposure monitoring elements requiring quality assurance (QA). Note that one must have a QA plan for many more factors than analytical techniques.

Figure A-8 shows a hypothetical example of an exposure distribution. In using an exposure distribution together with an exposure-response relationship to derive a quantitative risk estimate, one must decide to what population the exposure distribution should be applicable. Also whether the important exposure is the mean or one of the top percentiles must be decided.

ASSESSING DIOXIN EXPOSURE

In the early 1970s, a waste oil dealer in Missouri disposed of waste materials containing TCDD by mixing them with salvage oil and spraying the mixture on dirt roads and riding arenas. Measurements in soil gave values ranging from less than 1 to greater than 1,000 parts per billion (ppb) of TCDD. The Centers for Disease Control (CDC) was assigned the task of assessing possible health implications and, if feasible, recommending a soil concentration value for TCDD which should not be exceeded. The reference describing the results of CDC's deliberations is given below.

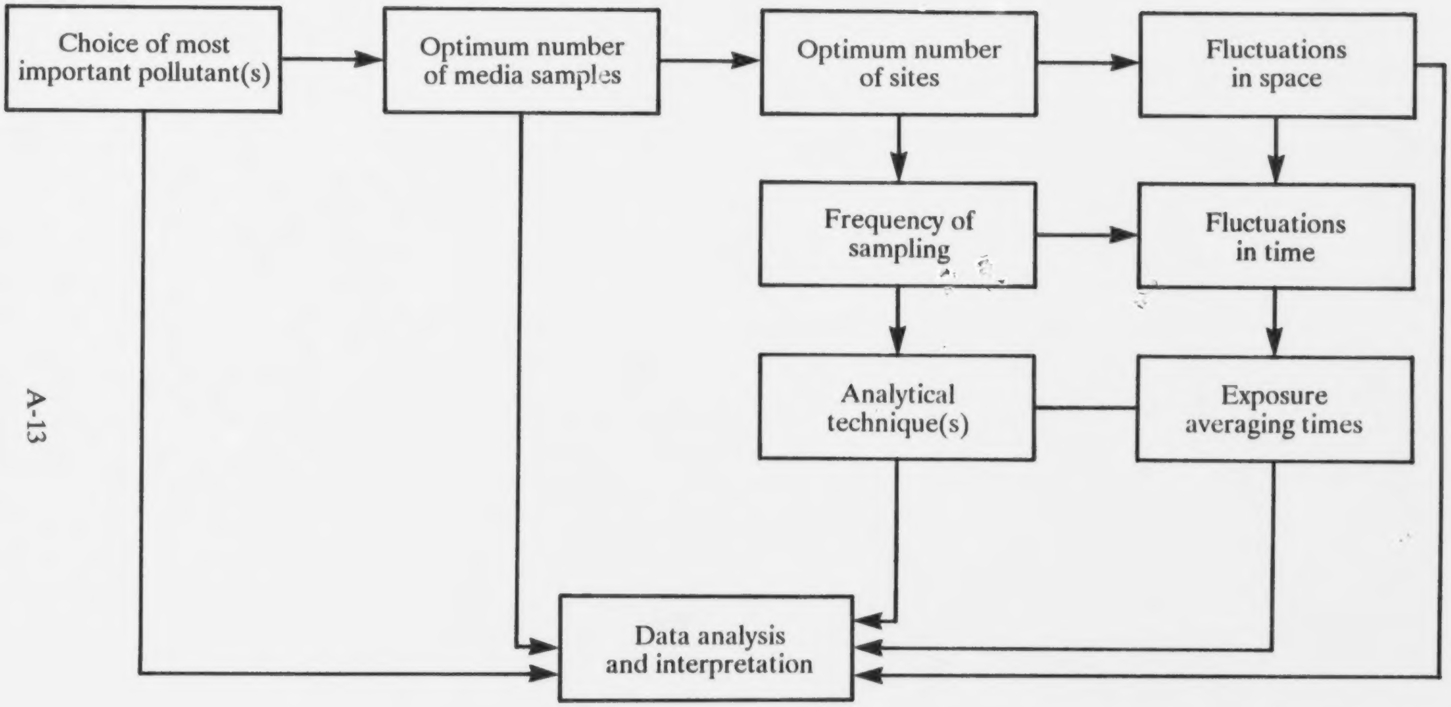

Choice of most important pollutant(s)
Optimum number of media samples
Optimum number of sites
Fluctuations in space
Frequency of sampling
Fluctuations in time
Analytical technique(s)
Exposure averaging times
Data analysis and interpretation

Figure A-7. Exposure monitoring elements requiring quality assurance.

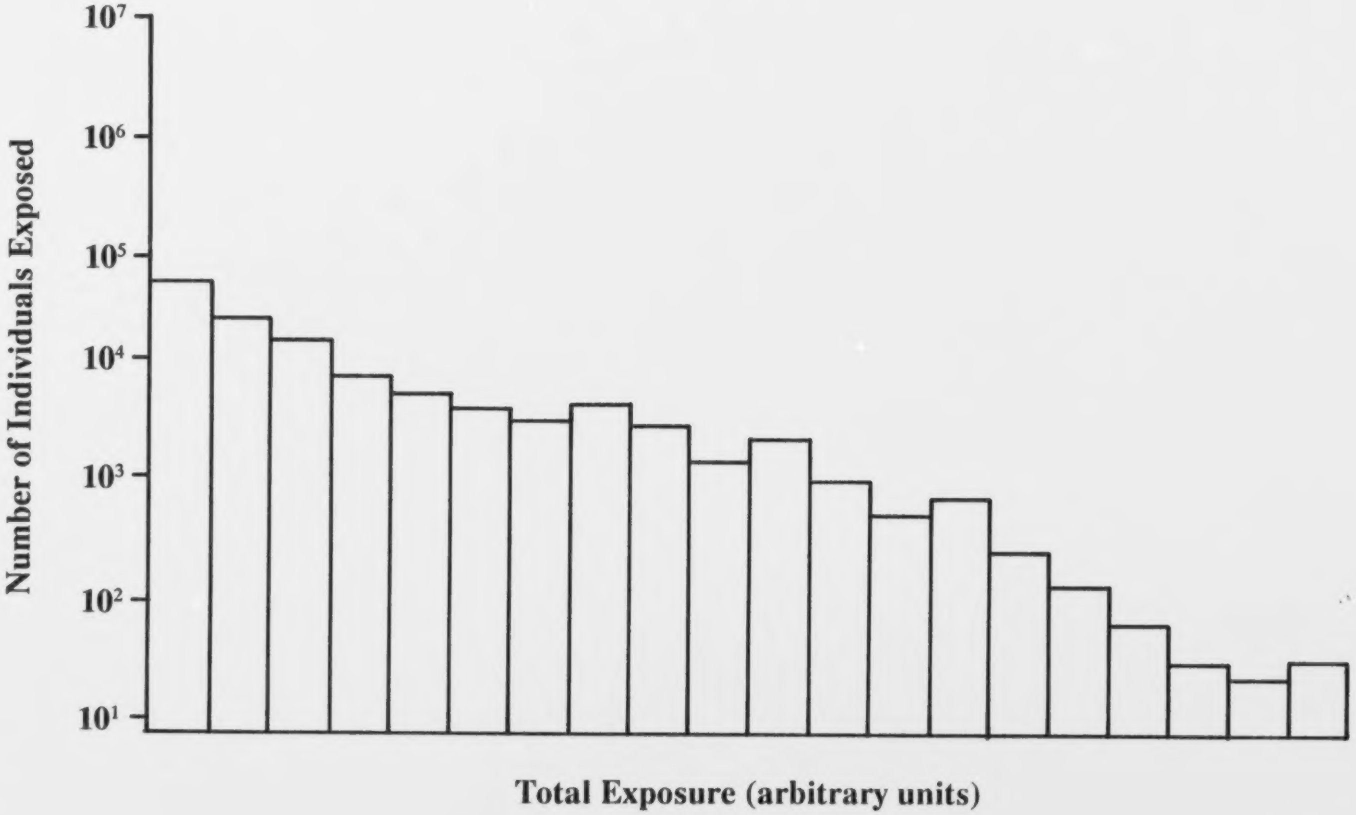


Figure A-8. Hypothetical exposure distribution.

Kimbrough, R.D., H. Falk, P. Stehr, and G. Fries. Health Implications of 2,3,7,8-Tetrachlorodibenzodioxin (TCDD) Contamination of Residential Soil. Jour. of Toxicol. and Environ. Hlth, 1984.

The following were identified as the most important factors influencing human exposure (dose):

- concentrations of environmental contamination,
- location of and access to contaminated areas,
- type of activities in contaminated areas,
- duration of exposure, and
- specific exposure mechanisms.

Figure A-9 shows the mathematical equation derived to calculate the total lifetime dose to TCDD. Note that dose is used rather than exposure. Exposure via a specific route may be converted to dose via the same route by multiplying the exposure by the percent absorbed. The use of exposure is more conservative since it is implicitly assumed that the absorption percent is 100. As an example, exposure to skin is the amount of pollutant in contact with the skin, whereas dose is the amount which is absorbed through the skin.

$$\text{Total Lifetime Dose} = \sum_{t=1}^{T} \text{TCDD}_t \times (\text{ING}_t \times \text{GI} + \text{DERM}_t \times \text{ABS} + \text{INH} \times \text{DUST}) \times \text{SEAS}$$

where T = expected lifespan (in days)

TCDD_t = concentration of TCDD in soil at time "t"

ING_t = age-specific amount of soil ingested at time "t"

GI = % absorbed through gastrointestinal tract

DERM_t = age-specific amount of soil deposited on skin at time "t"

ABS = % absorbed through skin

INH = amount of air exchanged per day

DUST = concentration of dust (from soil) in air

SEAS = "dummy" variable for seasonal access to outdoor contaminated areas (i.e., = 1 for fair weather months and = 0 for cold weather months)

Figure A-9. Equation for estimating total lifetime dose to TCDD.

Note that only three exposure routes were considered: dermal absorption through direct contact with the soil, ingestion of soil, and the inhalation of dust to which TCDD was attached. Possible exposures via inhalation of vapors or ingestion of food or water containing TCDD were not included. Probably the most serious omission is the food exposure route.

Some additional assumptions made on the basis of the limited data available are listed below:

- The environmental half-life of TCDD in soil is 12 years.
- TCDD levels in airborne dust are the same as those in soil.
- Indoor TCDD levels in dust are the same as outdoor levels.
- Fifteen m^3 of air is exchanged per person per day.
- The GI absorption rate of TCDD in soil is 30%.
- Exposures would take place only 6 months of the year because of seasonal influences and varying activity patterns.
- The dermal absorption rate of TCDD in soil is 1%.

Figure A-10 gives the estimated daily deposition of soil on skin by age. The same amounts were assumed to be ingested each day. These values are based on work done studying lead uptake from contaminated soils.

Age Group	Amount on Skin
0-9 months	0 grams
9-18 months	1 gram
1½-3½ years	10 grams
3½-15 years	1 gram
15 years	100 milligrams

Figure A-10. Estimated daily deposition of soil on human skin by age.

The authors make the point that their analysis applies only to residential areas and suggest that a lower safe value may be more appropriate for range or dairy farm areas, whereas a higher value may be adequate for commercial areas. Figure A-11 shows some results derived by the Food and Drug Administration (FDA) by analogy to polybrominated biphenyl (PBB) data. A maximum allowable intake of 100 picograms/day was assumed. Note that the value in soil which would produce the maximum allowable residue in milk is 6.2 pg/g or 6.2 parts per trillion (ppt).

Based on direct extrapolation of rodent data to humans and extrapolation to low doses by the linear derived multistage model, a dose of 28 fg/kg body weight/day is calculated as the virtually safe dose (an added cancer risk of $1/10^6$). For a 70 kg man, this is equivalent to 1.96 pg/person/day. A uniform concentration of 1 ppb in soil by the model used would lead to 44 pg/person/day.

Figure A-12 gives the estimated average daily dose corresponding to initial TCDD-soil contamination levels. It also shows the uncertainty ranges for both 10^{-6} and 10^{-5} excess lifetime cancer risks. On the basis of these results and the assumption that not 100% of contaminated areas would be at the peak level, the authors conclude that 1 ppb is a soil level of TCDD which should not be exceeded in residential areas.

Food	TCDD in fat, pg/g	Observed Ratio[1]	Soil, pg/g
Beef [2]	7.9	0.39	20
Beef (cull dairy)[3]	7.9	0.10	79
Pork	22.7	1.86	12
Milk	2.5	0.40	6.2

[1] Concentration of polybrominated biphenyls (PBB) in product/concentration of PBB in soil (Fries and Jacobs, 1983).

[2] Includes dairy cattle that have never lactated.

[3] Older cows. Younger cows would approach the values for beef cattle.

Figure A-11. Concentrations of TCDD in soil that are projected to produce the maximum allowable residues in foods.

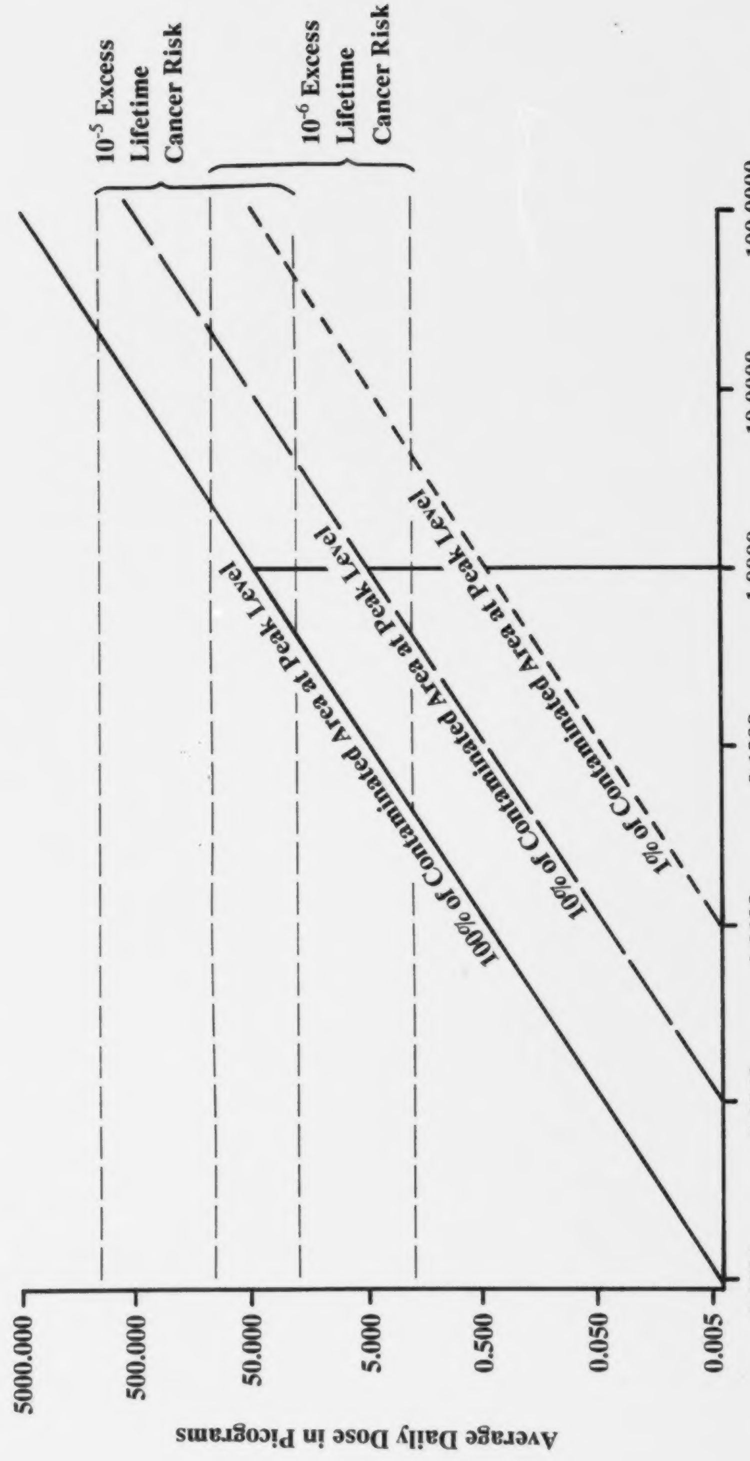


Figure A-12. Estimated average daily dose corresponding to initial TCDD-soil contamination levels.

APPENDIX B.

PERCENTILES OF THE t DISTRIBUTION

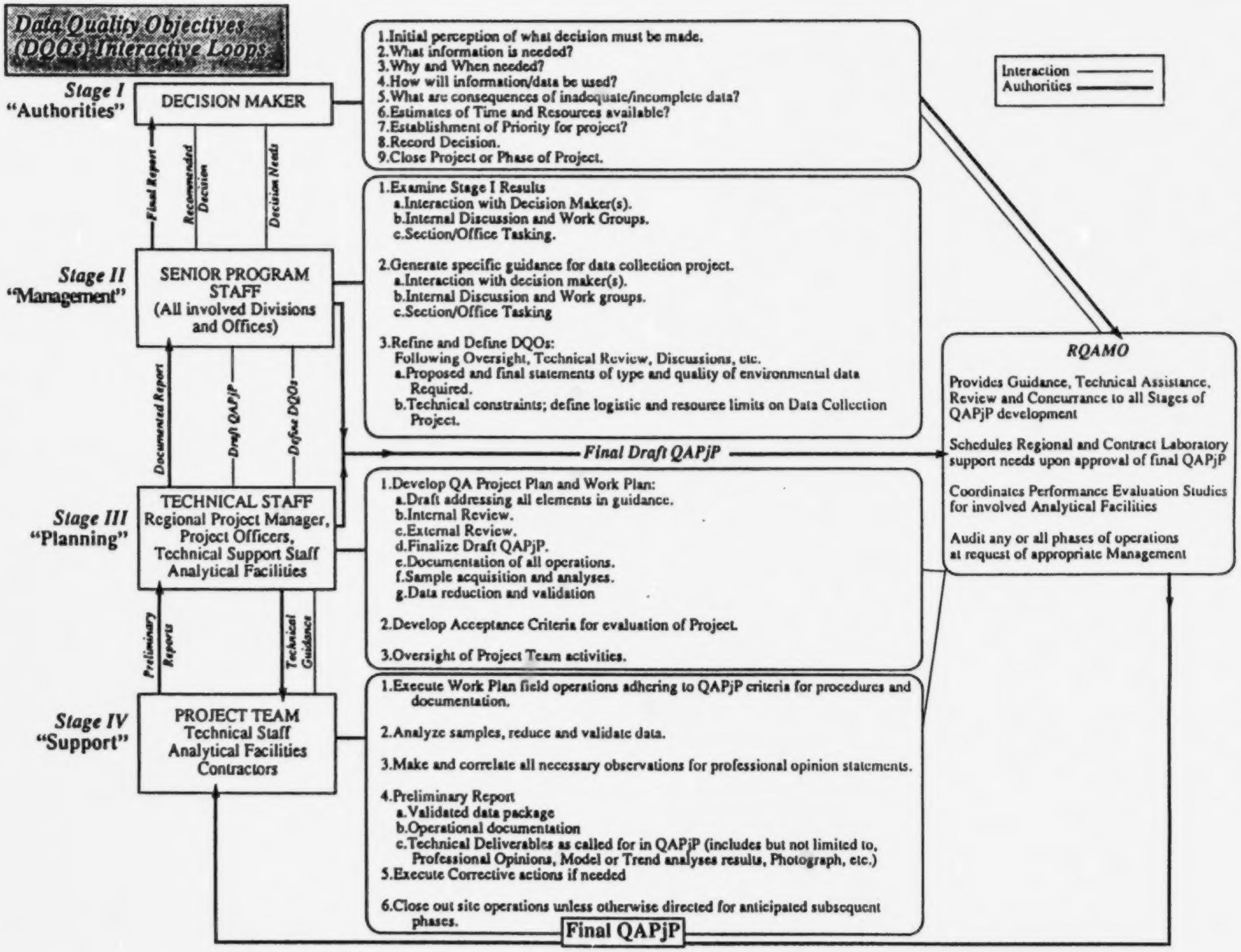
Confidence Level (%): 1-$\alpha/_2$ for two-tailed test

Confidence Level (%): 1-α for one-tailed test

	20	30	60	80	90	95	98	99
df	60	70	80	90	95	97.5	99	99.5
1	.325	.727	1.376	3.078	6.314	12.706	31.821	63.657
2	.289	.617	1.061	1.886	2.920	4.303	6.965	9.925
3	.277	.584	.978	1.638	2.353	3.182	4.541	5.641
4	.271	.569	.941	1.533	2.132	2.776	3.747	4.604
5	.267	.559	.920	1.476	2.015	2.571	3.365	4.032
6	.265	.553	.906	1.440	1.943	2.447	3.143	3.707
7	.263	.549	.896	1.415	1.895	2.365	2.998	3.499
8	.262	.546	.889	1.397	1.860	2.306	2.896	3.355
9	.261	.543	.883	1.383	1.833	2.262	2.821	3.250
10	.260	.542	.879	1.372	1.812	2.228	2.764	3.169
11	.260	.540	.876	1.363	1.796	2.201	2.718	3.106
12	.259	.539	.873	1.356	1.782	2.179	2.681	3.055
13	.259	.538	.870	1.350	1.771	2.160	2.650	3.012
14	.258	.537	.868	1.345	1.761	2.145	2.624	2.977
15	.258	.536	.866	1.341	1.753	2.131	2.602	2.947
16	.258	.535	.865	1.337	1.746	2.120	2.583	2.921
17	.257	.534	.863	1.333	1.740	2.110	2.567	2.898
18	.257	.534	.862	1.330	1.734	2.101	2.552	2.878
19	.257	.533	.861	1.328	1.729	2.093	2.539	2.861
20	.257	.533	.860	1.325	1.725	2.386	2.528	2.845
21	.257	.532	.859	1.323	1.721	2.080	2.518	2.831
22	.256	.532	.858	1.321	1.717	2.074	2.508	2.819
23	.256	.532	.858	1.319	1.714	2.069	2.500	2.807
24	.256	.531	.857	1.318	1.711	2.064	2.492	2.797
25	.256	.531	.856	1.316	1.708	2.060	2.485	2.787
26	.256	.531	.856	1.315	1.706	2.056	2.479	2.779
27	.256	.531	.855	1.314	1.703	2.052	2.473	2.771
28	.256	.530	.855	1.313	1.701	2.048	2.467	2.763
29	.256	.530	.854	1.311	1.699	2.045	2.462	2.756
30	.256	.530	.854	1.310	1.697	2.042	2.457	2.750
40	.255	.529	.851	1.303	1.684	2.021	2.423	2.704
60	.254	.527	.848	1.296	1.671	2.000	2.390	2.660
120	.254	.526	.845	1.289	1.658	1.980	2.358	2.617
oc	.253	.524	.842	1.282	1.645	1.960	2.326	2.576

APPENDIX C

DATA QUALITY OBJECTIVES DEVELOPMENT PROCESS


Data Quality Objectives (DQOs) Interactive Loops
Stage I "Authorities"
DECISION MAKER
Stage II "Management"
SENIOR PROGRAM STAFF (All involved Divisions and Offices)
Stage III "Planning"
TECHNICAL STAFF Regional Project Manager, Project Officers, Technical Support Staff Analytical Facilities
Stage IV "Support"
PROJECT TEAM Technical Staff Analytical Facilities Contractors
Final Report
Recommended Decision
Decision Needs
Documented Report
Draft QAPjP
Define DQOs
Preliminary Reports
Technical Guidance
Final Draft QAPjP
Final QAPjP
Interaction
Authorities
RQAMO

1.Initial perception of what decision must be made.
2.What information is needed?
3.Why and When needed?
4.How will information/data be used?
5.What are consequences of inadequate/incomplete data?
6.Estimates of Time and Resources available?
7.Establishment of Priority for project?
8.Record Decision.
9.Close Project or Phase of Project.

1.Examine Stage I Results
  a.Interaction with Decision Maker(s).
  b.Internal Discussion and Work Groups.
  c.Section/Office Tasking.

2.Generate specific guidance for data collection project.
  a.Interaction with decision maker(s).
  b.Internal Discussion and Work groups.
  c.Section/Office Tasking

3.Refine and Define DQOs:
  Following Oversight, Technical Review, Discussions, etc.
  a.Proposed and final statements of type and quality of environmental data Required.
  b.Technical constraints; define logistic and resource limits on Data Collection Project.

1.Develop QA Project Plan and Work Plan:
  a.Draft addressing all elements in guidance.
  b.Internal Review.
  c.External Review.
  d.Finalize Draft QAPjP.
  e.Documentation of all operations.
  f.Sample acquisition and analyses.
  g.Data reduction and validation

2.Develop Acceptance Criteria for evaluation of Project.

3.Oversight of Project Team activities.

1.Execute Work Plan field operations adhering to QAPjP criteria for procedures and documentation.

2.Analyze samples, reduce and validate data.

3.Make and correlate all necessary observations for professional opinion statements.

4.Preliminary Report
  a.Validated data package
  b.Operational documentation
  c.Technical Deliverables as called for in QAPjP (includes but not limited to, Professional Opinions, Model or Trend analyses results, Photograph, etc.)
5.Execute Corrective actions if needed

6.Close out site operations unless otherwise directed for anticipated subsequent phases.

Provides Guidance, Technical Assistance, Review and Concurrance to all Stages of QAPjP development

Schedules Regional and Contract Laboratory support needs upon approval of final QAPjP

Coordinates Performance Evaluation Studies for involved Analytical Facilities

Audit any or all phases of operations at request of appropriate Management

DATA QUALITY OBJECTIVES (DQOs) DEVELOPMENT CHECKLIST

FOR STAGE I (DECISION MAKER)

"AUTHORITIES"

1. Initial perception of what decision must be made. Complete? ☐

   Comment:____

2. What information is needed? Complete? ☐

   Comment:____

3. Why and When needed? Complete? ☐

   Comment:____

4. How will information/data be used? Complete? ☐

   Comment:____

5. What are consequences of inadequate/incomplete data? Complete? ☐

   Comment:____

6. Estimates of Time and Resources available? Complete? ☐

   Comment:____

7. Establishment of Priority for project? Complete? ☐

   Comment:____

8. Record Decision. Complete? ☐

   Comment:____

9. Close Project or Phase of Project. Complete? ☐

   Comment:____

DATA QUALITY OBJECTIVES (DQOs) DEVELOPMENT CHECKLIST

FOR STAGE II (SENIOR PROGRAM STAFF)

"MANAGEMENT"

1. Examine Stage I Results Complete? ☐
   a. Interaction with Decision Maker(s). ☐
   b. Internal Discussion and Work Groups. ☐
   c. Section/Office Tasking. ☐

   Comment:______________________________

2. Generate specific guidance for data collection project. Complete? ☐
   a. Interaction with decision maker(s). ☐
   b. Internal Discussion and Work groups. ☐
   c. Section/Office Tasking ☐

   Comment:______________________________

3. Refine and Define DQOs: Complete? ☐
   a. Proposed and final statements of type and quality of environmental data Required. ☐
   b. Technical constraints; define logistic and resource limits on Data Collection Project. ☐

   Comment:______________________________

DATA QUALITY OBJECTIVES (DQOs) DEVELOPMENT CHECKLIST

FOR STAGE III (TECHNICAL STAFF)

"PLANNING"

1. Develop QA Project Plan and Work Plan: Complete? ☐
    - a. Draft addressing all elements in guidance. ☐
    - b. Internal Review. ☐
    - c. External Review. ☐
    - d. Finalize Draft QAPjP. ☐
    - e. Documentation of all operations. ☐
    - f. Sample acquisition and analyses. ☐
    - g. Data reduction and validation ☐

   Comment:____

2. Develop Acceptance Criteria for evaluation of Project. Complete? ☐

   Comment:____

3. Oversight of Project Team activities. Complete? ☐

   Comment:____

DATA QUALITY OBJECTIVES (DQOs) DEVELOPMENT CHECKLIST

FOR STAGE IV (PROJECT TEAM)

"SUPPORT"

1. Execute Work Plan field operations adhering to QAPjP criteria for procedures and documentation. Complete? ☐

   Comment:____

2. Analyze samples, reduce and validate data. Complete? ☐

   Comment:____

3. Make and correlate all necessary observations for professional opinion statements. Complete? ☐

   Comment:____

4. Preliminary Report Complete? ☐

   Comment:____

   a. Validated data package ☐

   b. Operational documentation ☐

   c. Technical Deliverables as called for in QAPjP (includes but not limited to, Professional Opinions, Model or Trend analyses results, Photograph, etc.) ☐

5. Execute Corrective actions if needed Complete? ☐

   Comment:____

6. Close out site operations unless otherwise directed for anticipated subsequent phases. Complete? ☐

   Comment:____

EXAMPLE FORMAT AND CRITICAL ELEMENTS OF

QUALITY ASSURANCE PLAN

Project name:____________________
Project code:____________________
Address:________________________
____
Responsible organization:__________
____

Approvals:

Project Officer:____________________________Date________
QA Officer:________________________________Date________
ESD Peer Review:____________________________Date________
Regional Sample Control Center (RSCC):______________Date________
Supervisor:________________________________Date________

- PROJECT ORGANIZATION AND RESPONSIBILITY

The following is a list of key project personnel and their responsibilities:

Organization Manager ______________________________
Project Officer ______________________________
QA Officer ______________________________
Field Operation ______________________________
Laboratory Operation ______________________________
Data Quality Review ______________________________
System/Performance Audit ______________________________

- PROJECT CODES AND SAMPLE NUMBERS (to be completed by RSCC)

Project NO.:____________________ Account NO.:____________________
Laboratory Designated:____________ EPA__________ CLP__________ Private
Sample Numbers assigned: from____________________ to____________________

- PROJECT DESCRIPTION

1. Objective and Scope:______________________________

____

____

2. Schedule of Tasks and Milestones:

	Dates				
Activities					

3. Data Usage:______________________________

____

____

4. Monitoring network/sample collection design and rationale: ____________

____

____

- PROJECT DESCRIPTION - continued

# of Samples	Sample Matrix	Collection Frequency	Analytical Parameter	Type of Sample Container	Sample Preservation	Holding Time	Analytical Detection Limit	Quality Control Samples

- DATA QUALITY OBJECTIVES

1. Precision and Accuracy protocols/limits: ____________________

2. Data Representativeness: ____________________

3. Data Comparability: ____________________

4. Data Completeness: ____________________

- SAMPLING PROCEDURES (including QC checks):

- SAMPLE CUSTODY PROCEDURES:

- CALIBRATION PROCEDURES AND PREVENTIVE MAINTENANCE:

- ANALYTICAL METHODS (including QC checks):

- DOCUMENTATION, DATA REDUCTION AND REPORTING

  1. Documentation:

  2. Data Reduction and Reporting:

- DATA ASSESSMENT:

- PERFORMANCE/SYSTEM AUDITS:

- CORRECTIVE ACTION:

____

____

- REPORTS:

____

____

SAMPLE ALTERATION CHECKLIST

Project Name and Number:

Material to be sampled:

Measurement Parameter:

Standard Procedure for Field collection & Laboratory Analysis (cite references):

Reason for change in Field Procedure or Analytical Variation:

Variation from Field or Analytical Procedure:

Special Equipment, Materials, or Personnel Required:

Initiators Name: ______________________ Date: ______________________

Project Approval: ______________________ Date: ______________________

Laboratory Approval: ______________________ Date: ______________________

QA Officer/Reviewer: ______________________ Date: ______________________

Sample Control Center: ______________________ Date: ______________________

CORRECTIVE ACTION CHECKLIST

Project Name and Number: ____________________

Sample Dates Involved: ____________________

Measurement Parameter(s): ____________________

Acceptable Data Range: ____________________

Problem Areas Requiring Corrective Action: ____________________

Measures Required to Correct Problems: ____________________

Means of Detecting Problems and Verifying Correction: ____________________

Initiators Name: ____________________ Date: ____________________

Project Approval: ____________________ Date: ____________________

Laboratory Approval: ____________________ Date: ____________________

QA Officer/Reviewer: ____________________ Date: ____________________

Sample Control Center: ____________________ Date: ____________________